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INVESTIGATION OF THE EFFECT OF MOISTURE ON THE MECHANICAL PROPERTIES OF ORGANIC MATRIX COMPOSITE MATERIALS USING NUCLEAR MAGNETIC RESONANCE

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G. A. MATZKANIN
Southwest Research Institute
6220 Culebra Road, P.O. Drawer 28510
San Antonio, Texas 78284

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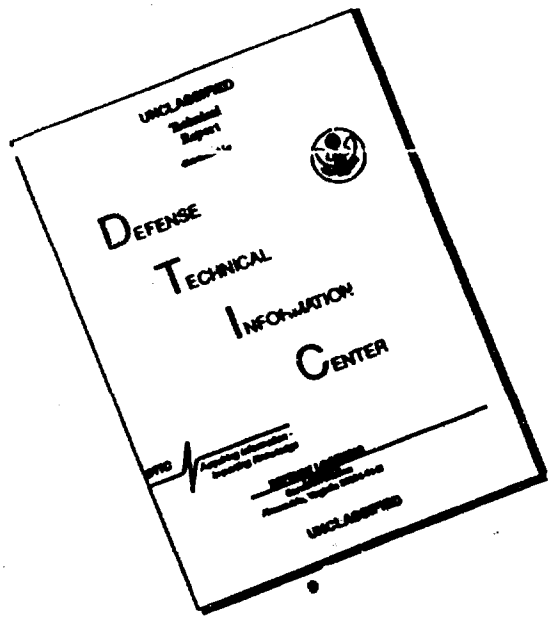

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Investigations were conducted of nuclear magnetic resonance (NMR) signals from hydrogen atoms in two organic matrix composite systems subjected to environmental conditioning at 51.6°C (125°F) and 95% relative humidity. The composite systems were 8 ply, ±45° laminates fabricated from SP 250 resin/S2 glass fiber and Reliabond 9350 resin/Kevlar 49 fiber. Absorbed moisture free induction decays consisted of distinct multiple components attributed to moisture in various states of molecular binding. →cont

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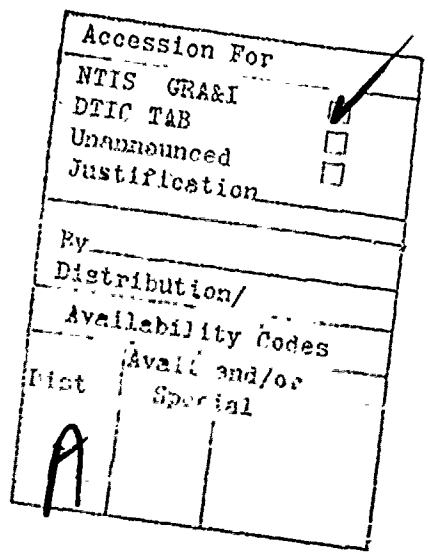
Plasticizing of the resin matrix resulted in an increase in nuclear relaxation time with increasing moisture. Good correlation was obtained between the NMR signal amplitude and dry weight moisture percentage for both composite systems. Destructive tensile tests were performed on dried specimens and on conditioned specimens at levels of 4.6% and 1.3% dry weight moisture for the Kevlar and glass composites, respectively. Reductions in ultimate tensile strength due to moisture conditioning were 14% for the Kevlar composite and 4.3% for the glass composite.

PREFACE

This project was accomplished as part of the U.S. Army Aviation Research and Development Command Manufacturing program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel. Comments are solicited on the potential utilization of the information contained herein as applied to present and/or future production programs. Such comments should be sent to: U.S. Army Aviation Research and Development Command, ATTN: DRDAV-EGX, 4300 Goodfellow Blvd., St. Louis, MO 63120.

The work described in this report was accomplished under a contract monitored by the Army Materials and Mechanics Research Center. Technical monitor for this contract was Dr. R. J. Shuford.



FOREWORD

This project was performed by Southwest Research Institute for the U.S. Army Materials and Mechanics Research Center as a special task under auspices of the Nondestructive Testing Information Analysis Center (NTIAC). Funding was provided through NTIAC under Item No. 0001AG of Contract DLA 900-79-C1266. Appreciation is expressed to Mr. Richard Shuford of the Composites Development Division at AMMRC for his technical guidance throughout the performance of this program, to the Contracting Officers Technical Representative, Mr. George Darcy for his cooperation in arranging this study and to Dr. Richard Smith, Director of NTIAC.

The Task Manager for this program was G. A. Matzkanin of the Instrumentation Research Division at Southwest Research Institute; much of the NMR experimental work was performed by E.S. Reiwerts. Mechanical testing was supervised by R. D. Brown of the Department of Materials Sciences.

EXECUTIVE SUMMARY

This project was undertaken to investigate the feasibility of using nuclear magnetic resonance (NMR) to nondestructively determine the amount of moisture in organic matrix composites and the extent of moisture-induced mechanical degradation.

The investigations consisted of measuring the hydrogen NMR signals from two organic matrix composite systems subjected to long term environmental conditioning. The composite systems investigated were 8 ply, ± 45° laminates fabricated from SP 250 resin/S2 glass fiber and Reliabond 9350 resin/Kevlar 49 fiber. Specimen coupons, 20.3 cm (8 in.) X 2.54 cm (1 in.), were cut from the panels and initially oven-dried before conditioning at 51.6°C (125°F) and 95% relative humidity for approximately four months. Destructive tensile testing was performed on ten dried specimens and ten conditioned specimens after they had reached levels of 4.6% and 1.3% dry weight moisture for the Kevlar and glass composites, respectively. Moisture absorption was found to cause decreases in ultimate tensile strength of 14% and 4.3% for the Kevlar and glass composites, respectively, and an increase in the tangent modulus of approximately 11% for both types of composite.

Although the ultimate sensitivity and resolution were not determined, absorbed moisture levels as low as 0.2% produced measureable NMR signals. The absorbed moisture NMR signals consisted of distinct multiple components which were attributed to moisture in various states of molecular binding. Good correlation was obtained between the NMR signal amplitude and the dry weight moisture percentage indicating the potential feasibility of utilizing NMR for nondestructively measuring the amount of moisture in composites. Analysis of the NMR signals indicated that plasticizing of the resin matrix was the most likely cause of mechanical degradation rather than microcracking. Experiments on fractured specimens subjected to additional environmental exposure showed that NMR is capable of distinguishing the free moisture entering a composite through cracks and fissures from that absorbed into the composite structure. Experiments performed on fibers indicated that NMR is capable of detecting and measuring moisture absorbed by Kevlar fiber. As expected, no hydrogen NMR signal was observed from the glass fiber.

Based on the results of this project, it is recommended that additonal NMR investigations be pursued to investigate the physical mechanisms of moisture absorption by composites and the role played by moisture in mechanical degradation. Specific experiments suggested include the measurement of NMR parameters such as the spin-lattice relaxation time and a study of the effect of applied stress on moisture absorption.

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I. INTRODUCTION

Moisture intrusion caused by environmental exposure for extended periods is known to have an adverse effect on the structural integrity of organic matrix composite materials.[1] Not only does moisture migration along the fiber-matrix interface weaken the interface bond, but also diffusion through the organic matrix leads to degradation of the polymeric structure. Both interactions result in a decrease in mechanical properties of the composite.

The usual method of determining moisture absorption in the laboratory is by means of weight gain measurements. While this method is satisfactory for laboratory use, it is not practical for field measurements. The work reported herein, was undertaken to investigate the feasibility of using nuclear magnetic resonance (NMR) as an alternative method for determining the amount of moisture in organic matrix composites. NMR is a rapid, entirely nondestructive spectroscopic approach involving the resonant absorption of electromagnetic energy by nuclei with magnetic moments. The NMR method has been used extensively as an analytical laboratory tool in physics and chemistry. In addition, instrumentation systems have been developed and applied to a number of difficult but practical moisture measurement problems including moisture measurement in black powder and smokeless powder, corn and potato food products, and highway subgrade soils.[2,3,4] One of the important features of NMR is that not only can the amount of moisture be determined but the method can also distinguish between water in different binding states, thus providing information on the molecular structure of the material. In the experiments performed on composites, the NMR signals were examined to determine the feasibility of associating certain signal characteristics with mechanical degradation caused by moisture.

As described in the next section on experimental procedure, two composite specimen systems were investigated. One, a fiberglass-based system and the other a Kevlar fiber-based system. The specimens were conditioned in an environmental chamber at 51.6°C (125°F) and 95% relative humidity. NMR measurements were made several times during the moisture absorption cycle and the signals analyzed in terms of the amount of moisture present. Mechanical property measurements were made on oven-dried specimens and on moisture-saturated specimens. Results of the experiments presented in Section III show that the NMR signals are composed of several components which can be associated with moisture in different states of binding. The moisture component of the NMR signal correlates well with the amount of moisture determined by weighing and demonstrates that NMR is a sensitive and accurate method for nondestructively determining the amount of moisture present in composites. Based on the measurements made, however, the association between NMR signal characteristics and mechanical degradation is not clear. Additional measurements are needed, for example, on the fiber and resin component materials, in order to better understand the composition of the NMR signals, and their relationship to the physical and molecular structure of the composite. Based on the results obtained it is clear that NMR is a useful indicator of moisture content in organic composites and in addition has the capability for providing information on molecular structure changes accompanying environmental exposure.

During the performance of the project, a literature survey was conducted on methods for measuring moisture in composites and the effects of moisture absorption on mechanical properties. Because of the importance of this topic to the NDE and mechanical design communities, the literature survey conducted during this project will be expanded into a state-of-the-art survey under sponsorship of the Nondestructive Testing Information Analysis Center (NTIAC) and will appear in the near future as an NTIAC publication available for public distribution. Results of the literature search are briefly described in Appendix A to this report.

II. EXPERIMENTAL PROCEDURE

Specimen Conditioning

The composite specimens utilized in this study were of two types. One type was composed of SP 250 resin and S2 glass fibers. The other type was composed of Reliabond 9350 resin and Kevlar 49 fibers. These two types of composite materials were used to fabricate 8-ply laminates with a $\pm 45^{\circ}$ fiber orientation. Test specimens of nominal dimensions, 2.54 cm (1-in.) by 20.3 cm (8-in.), were cut from the laminate panels. Twenty-two specimens of each type were cut for the testing program, the glass fiber specimens labeled G1-G22 and the Kevlar fiber specimens labeled K1-K22. The specimens were left untabbed to ensure uniform moisture conditioning.

Fiber volumes for the two composite systems were determined by taking a sample of the composite, weighing it first in air then in water to determine the panel density as described in ASTM D792 "Specific Gravity and Density of Plastics by Displacement". Fiber volume was then calculated from the densities by the equation:

$$V_F = \frac{\rho_C - \rho_R}{\rho_F - \rho_R} \times 100\%$$

where ρ is density; subscript C refers to the composite, R refers to resin, and F refers to fiber. Void volume for the glass composite was determined by the method described in ASTM D2584 "Ignition and Loss of Cured Reinforced Resin". Void volume could not be determined for the Kevlar composite due to disintegration of the Kevlar fiber during the ignition process. Results for the two composite systems are tabulated in Table 1.

Prior to moisture conditioning, all 44 composite specimens were oven-dried at a temperature of $55^{\circ}C$ ($131^{\circ}F$) in a vacuum oven. Aluminum racks were fabricated to keep the specimens separated from one another in the oven. The specimens were all weighed prior to placement in the oven and five specimens of each type (glass and Kevlar) were removed from the oven and weighed periodically to determine when an equilibrium condition was reached. All weighings were performed on a Mettler balance capable of 0.0001 gram resolution. A constant dry weight condition for the composite specimens was achieved after three weeks of oven drying.

Upon completion of oven drying, ten each of the two types of specimens (G13-G22 and K13-K22) were destructively tested to determine the dry mechanical properties. Of the remaining oven dried specimens, NMR measurements were performed on specimens G1-G5 and K1-K5. Specimens G1, G12, K1 and K12 were placed in a desiccator and kept in a dried condition. These specimens were utilized throughout the remainder of the project as baseline specimens to check the calibration of the NMR instrumentation system. The

TABLE 1

Material Parameters of Composite Specimens

	Kevlar Composite	Glass Composite
Resin Type	Reliabond 9350	SP 250
Fiber Type	Kevlar 49 Fabric	S2 Glass
Resin Density	1.20 g/cm^3	1.21 g/cm^3
Fiber Density	1.45 g/cm^3	2.49 g/cm^3
Composite Density	1.33 g/cm^3	1.80 g/cm^3
Fiber Volume	47.9%	47.5%
Void Volume	---	1.26%

remaining twenty specimens (G2-G11 and K2-K11) were placed in the environmental chamber for moisture conditioning. A summary of specimen disposition is given in Table 2.

Moisture conditioning was accomplished under constant temperature-humidity conditions using a commercially manufactured controlled environmental chamber (Tenny Engineering Inc., Union New Jersey). With this chamber, heat is suppied by resistance heating elements and moisture is supplied by injecting steam into the cabinet. During environmental exposure, humidity was measured and controlled from wet and dry bulb temperature measurements. Environmental conditioning was performed at a temperature of 51.6°C (125°F) and a relative humidity of 95%. The specimens in the chamber were held separated from one another on the same racks used for oven drying.

Moisture absorption within the specimens was determined by the change in weight of the specimens. During moisture conditioning, three to six specimens of each type were periodically removed from the environmental chamber and weighed. A typical measurement resulted in at most a ten minute period during which the specimens were exposed to room temperature conditions. The particular specimens removed for weighing were varied so that the same specimens were not removed from the chamber each time. During the first two weeks of conditioning, specimens were weighed daily with the interval increasing to weekly after this initial period.

NMR Measurements

NMR measurements were performed on the composite specimens utilizing a 30 MHz pulsed NMR spectrometer. The basic principles of the NMR method are described in Appendix B and the references cited therein. Briefly, application of NMR to composite materials involves measurement of the hydrogen nucleus (proton) NMR signal. The experimental procedure involves placing the specimen into a radiofrequency (RF) coil which is located within the gap of an electromagnet (see Figure B7, Appendix B). The RF coil utilized for the measurements on composites was approximately 3.2 cm (1.25 in.) long and had an inner diameter slightly greater than the width of the specimens. The coil mounting assembly was constructed of hydrogen-free materials and measurements verified that no hydrogen NMR signal was observed from the empty coil.

The principal NMR signal parameter measured was the free induction decay (FID) following a 90°RF pulse (see Figure B4, Appendix B). The FID allows a determination of the nuclear spin-spin relaxation time T_2 which provides information on molecular coupling and nuclei in different chemical states. To improve the signal-to-noise ratio of the measurements, signal averaging was utilized. This involved repetitive sampling of the NMR signal and time averaging to reduce the noise level relative to the signal amplitude. For signal averaging, a Biomation Model 8100 Transient Recorder was used in conjunction with a Nicolet 1072 Instrument Computer. The time averaged NMR signals were displayed on a Moseley XY Recorder for later analysis.

TABLE 2

Disposition of Composite Specimens

All specimens initially oven-dried

Kevlar Composite Specimens	Specimen Utilization	Glass Composite Specimens
K1	Kept dry in desiccator; NMR	G1
K2	NMR measurements during moisture conditioning	G2
K3		G3
K4		G4
K5		G5
K6	Moisture conditioned; destructively tested after moisture saturation	G6
K7		G7
K8		G8
K9		G9
K10		G10
K11		G11
K12	Kept dry in desiccator	G12
K13	Destructively tested after oven-drying	G13
K14		G14
K15		G15
K16		G16
K17		G17
K18		G18
K19		G19
K20		G20
K21		G21
K22		G22

During the moisture conditioning period, NMR measurements were performed on four specimens of each type (G2-G5 and K2-K5) at several levels of moisture absorption. For these measurements, the specimens were removed from the environmental chamber and placed into the RF coil of the NMR system. The composite specimens were weighed both before and after the NMR measurements and accurate records kept of the specimen weights so that any extraordinary change in moisture level would be noted. The average time a specimen was out of the environmental chamber during NMR measurements was approximately 30 minutes with the change in dry weight moisture percentage during this time typically being less than 0.05% for the Kevlar specimens and less than 0.01% for the glass specimens.

Mechanical Testing

Destructive mechanical testing was performed on oven-dried specimens G13-G22 and K13-K22. Upon completion of moisture conditioning and after performing final NMR measurements, environmentally conditioned specimens G2-G11 and K2-K11 were also subjected to destructive mechanical testing. Mechanical testing consisted of determining tensile strength and modulus using a 222.4 kN (50 kip) hydraulic test machine with a head displacement rate of 0.13 cm (0.05-in.) per second. Strain was measured by means of a strain gage extensometer having a nominal 5.1 cm (2-in.) gage length. The stress-strain curve for each specimen was recorded using an XY recorder and the modulus and ultimate strength determined from these data.

III. RESULTS

Moisture Absorption

Moisture saturation took considerably more time than anticipated, possibly due to the low fiber volumes for these composites (see Table 1). In fact, complete equilibrium moisture saturation was not attained within the time frame of the project as can be seen from the moisture absorption curves shown in Figures 1 and 2 for the Kevlar and glass fiber composite systems, respectively. The data are shown plotted in the usual format of dry weight moisture percentage versus $\sqrt{\text{time}}$.[5] After a little over four months of environmental exposure, the Kevlar composite specimens reached a moisture level of 4.6% while the glass composite specimens reached a level of 1.3%. In order to complete the project in a timely manner, it was decided to perform the final NMR and mechanical testing experiments at these moisture levels even though the specimens were not at saturation equilibrium. All of the moisture levels at which NMR measurements were made are noted on the moisture absorption curves. As indicated previously, the moisture percentage values were obtained by weighing three to six specimens of each type. The weights among specimens were found to be quite consistent and in practically every case, the weights were within the extent of the data points on the graphs in Figures 1 and 2.

After three months of moisture conditioning, the environmental chamber malfunctioned resulting in a loss of humidity generation for a period of approximately 24 hours. During the time that the chamber was undergoing repair, the composite specimens were put into sealed containers and placed in an oven at a temperature of 51.6°C (125°F) in order to simulate conditions in the environmental chamber. As seen in Figures 1 and 2, the chamber malfunction resulted in a loss in moisture of approximately 0.1% dry weight moisture percentage for both the Kevlar and glass composite specimens. After the environmental chamber was repaired, moisture reabsorption occurred quickly so that by the time final NMR and mechanical measurements were made, the moisture levels in the specimens coincided with the original moisture absorption curve.

NMR Measurements

Typical NMR free induction decay signals from wet and dry Kevlar and glass composite specimens are shown in the oscilloscope photographs in Figures 3, 4, 5, and 6. In these figures, the oscilloscope vertical (V) and horizontal (H) sensitivities are indicated as well as the attenuator (Atten) setting of the NMR spectrometer. The bottom photographs in these figures present the same information as the top photographs but at greater vertical sensitivity and an expanded time base in order to better display the moisture component. In Figure 3 which shows results for a Kevlar composite specimen containing 4.5% moisture two components in the FID can be readily distinguished. The large amplitude, fast decaying component identified as

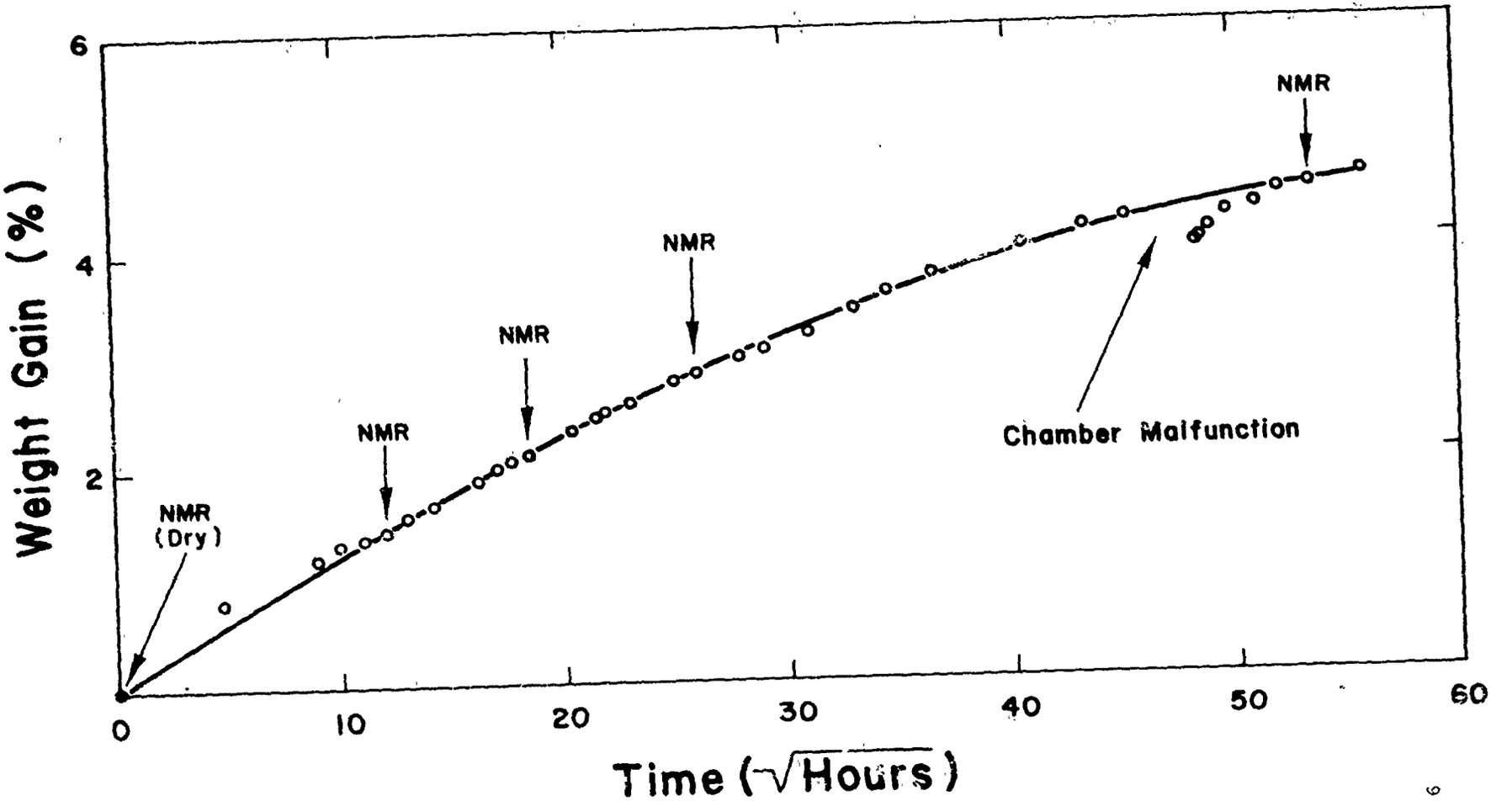


FIGURE 1. MOISTURE ABSORPTION FOR KEVLAR COMPOSITE

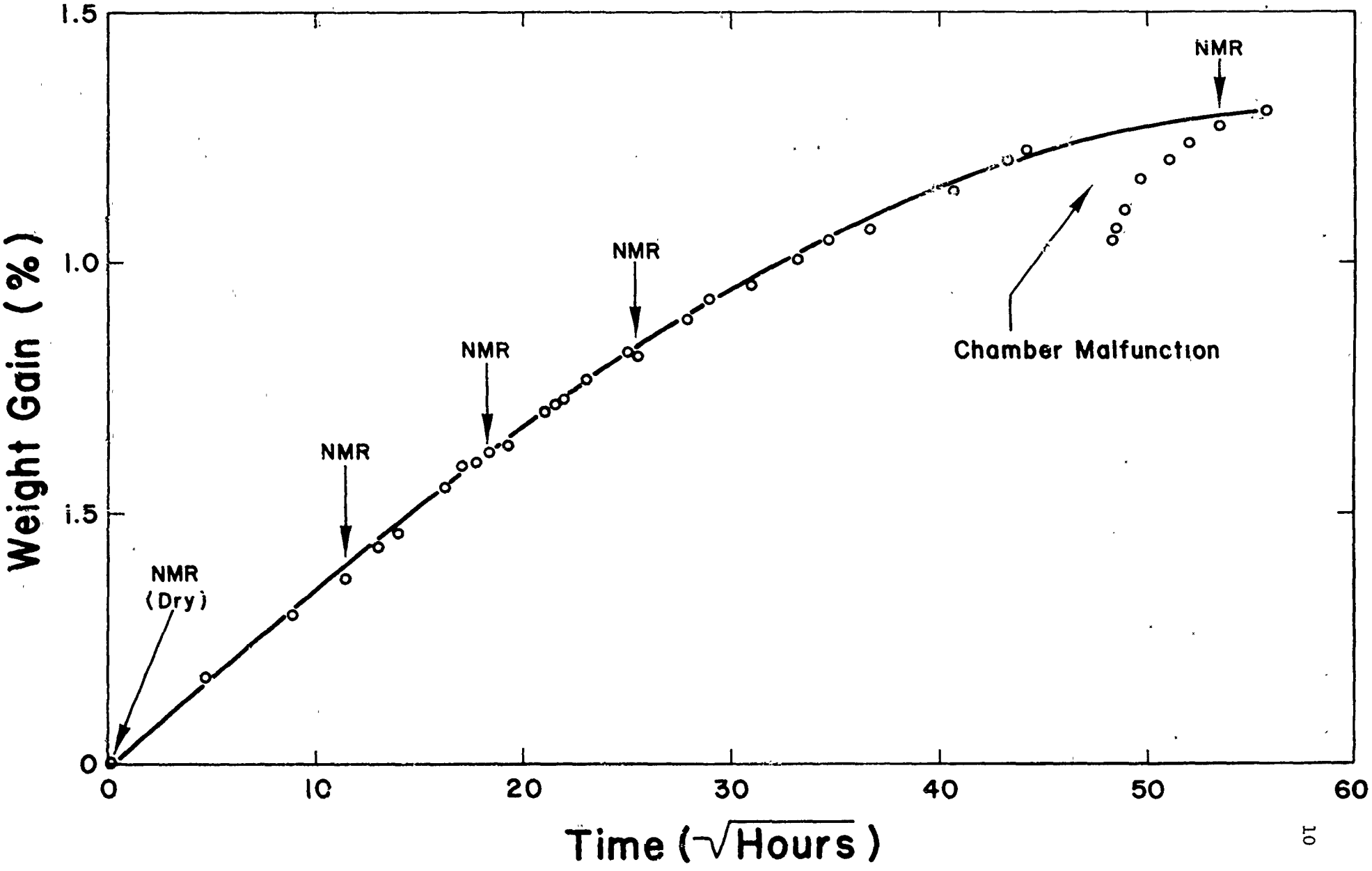
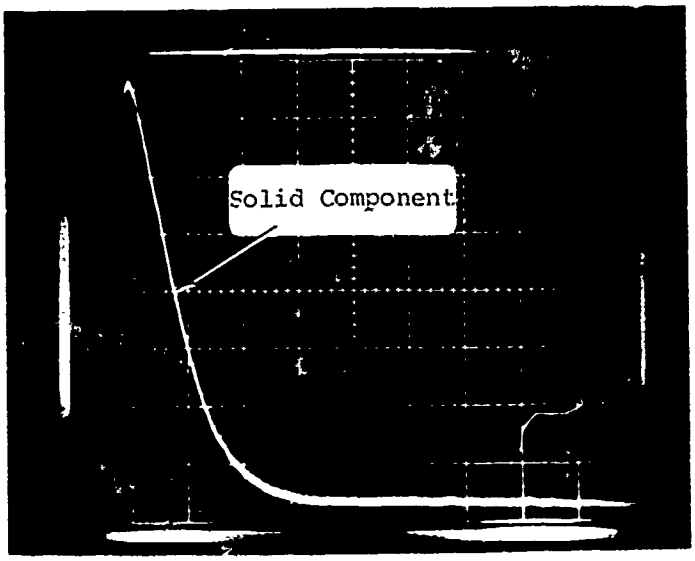


FIGURE 2. MOISTURE ABSORPTION FOR GLASS COMPOSITE



(a)

V - 0.2 V/div

H - 10 μs/div

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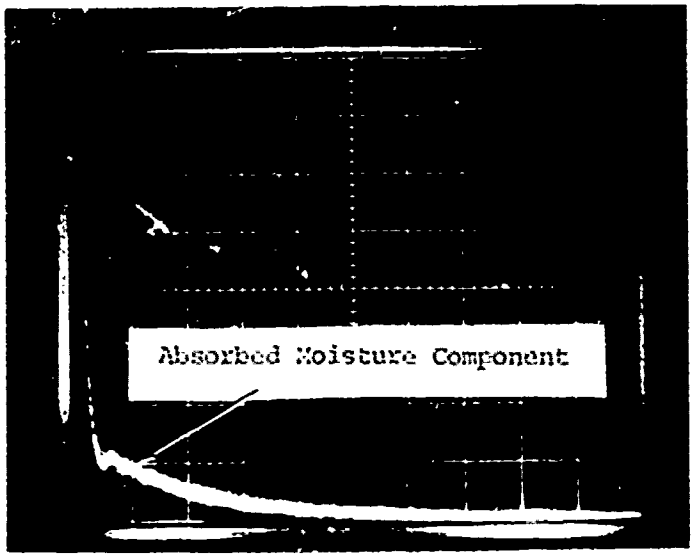

(b)

V - 0.05 V/div

H - 100 μs/div

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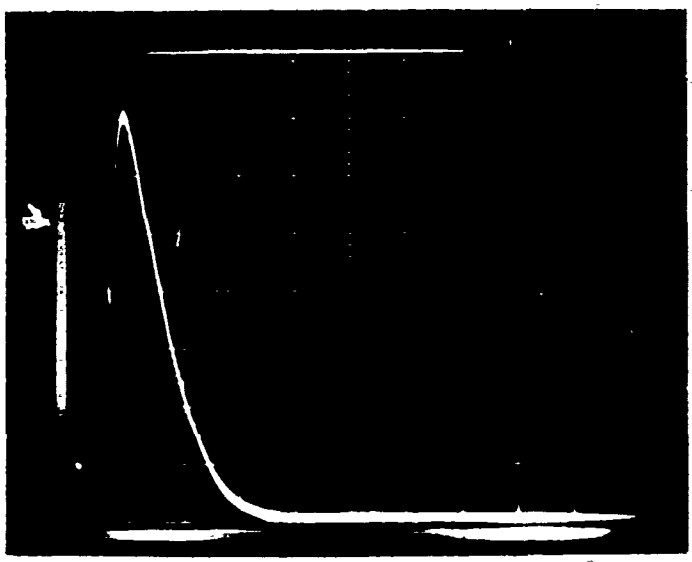
FIGURE 3. FREE INDUCTION DECAYS FROM KEVLAR FIBER COMPOSITE WITH 4.6% MOISTURE



(a)

V - 0.2 V/div

H - 10 μs/div

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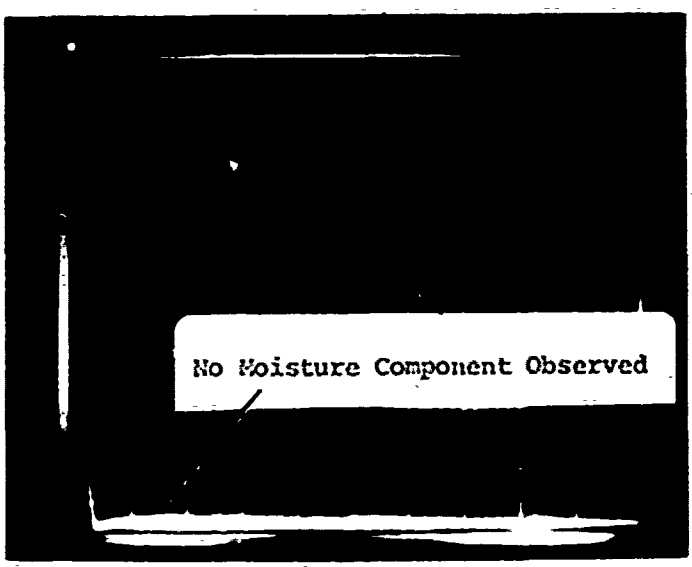

(b)

V - 0.05 V/div

H - 100 μs/div

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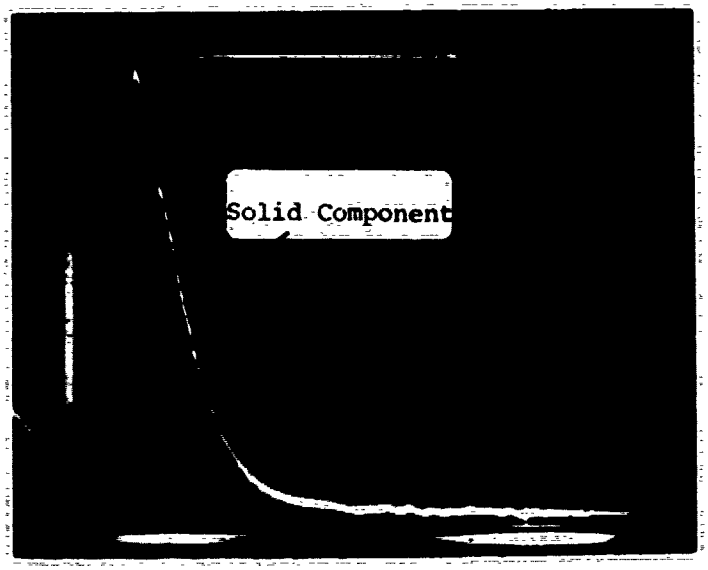
FIGURE 4. FREE INDUCTION DECAYS FROM OVEN-DRIED KEVLAR FIBER COMPOSITE

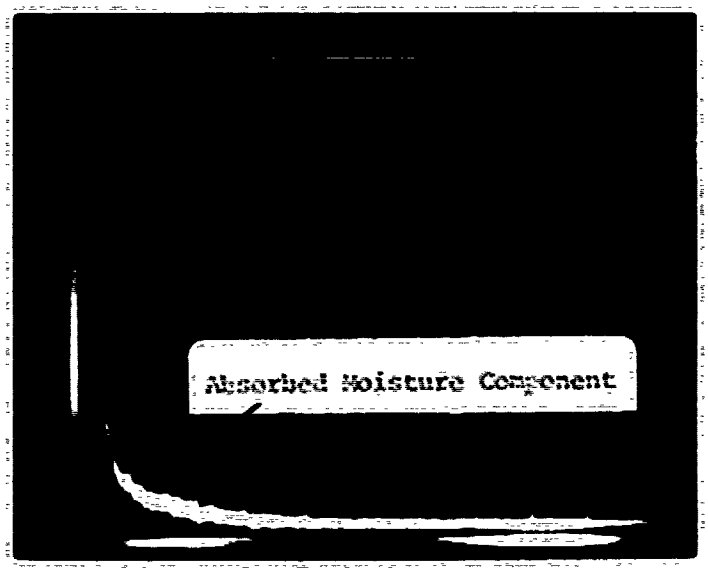


(a)

V = 0.1 V/div

H = 10 μs/div

Atten: - 28 DB



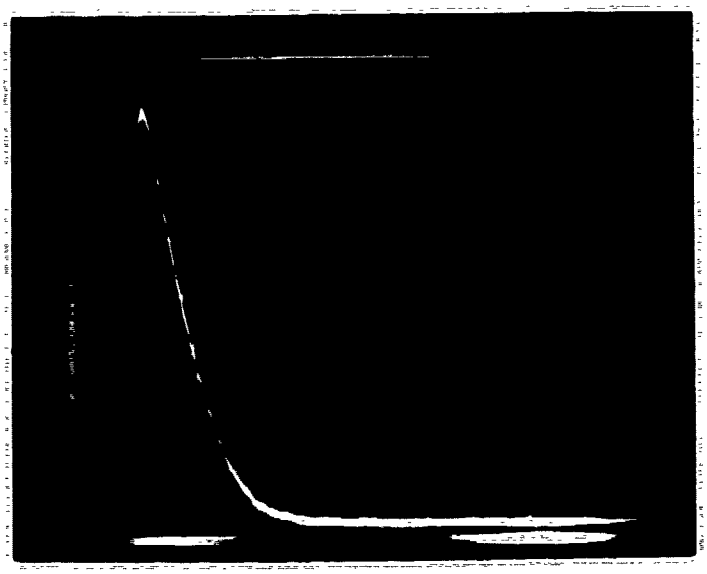
(b)

V = 0.02 V/div

H = 100 μs/div

Atten: - 28 DB

FIGURE 4. PULSE ISOLATION SIGNAL FROM GLASS FIBER COMPOSITE WITH LOW % LIMIT



(a)

V = 0.1 V/div

H = 10 μs/div

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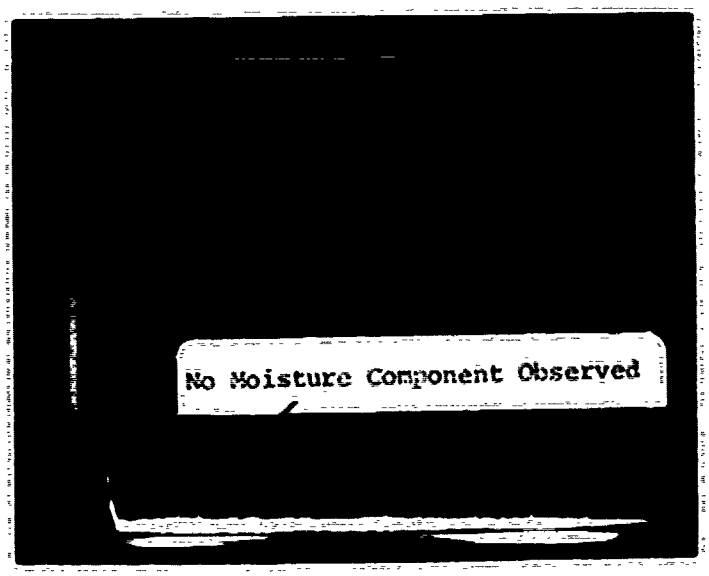


(b)

V = 0.02 V/div

H = 100 μs/div

Atten: = 28 DB

FIGURE 6. FREE INDUCTION DECAYS FROM OVEN-DRIED GLASS FIBER COMPOSITE

"Solid Component" in Figure 3a is associated with the chemically bound structural protons in the Kevlar composite material. The other lower-amplitude FID component shown more clearly on the expanded scale photograph in Figure 3b is associated with the absorbed moisture. (As will be shown later in this section, the moisture signal is actually composed of multiple components associated with moisture in different states of molecular binding). For comparison, the FID obtained from an oven-dried Kevlar composite specimen is shown in the oscilloscope photographs in Figure 4. Note that even in the expanded scale photograph in Figure 4b, there is no evidence of a moisture component (compare Figures 3b and 4b). Comparable results for wet and dry glass composite specimens are shown in Figure 5 and 6, respectively. The lower amplitude for the glass composite moisture component in Figure 5b compared with that for the Kevlar composite in Figure 3b (note the change in vertical sensitivity) is due to the lower moisture content in the glass composite relative to the Kevlar composite.

In order to quantitatively analyze the NMR signals from the composites, the time averaged FID's were measured from the XY recordings and plotted on semi-log graph paper. Since the nuclear spin relaxation times are expected to be exponential (see Appendix B), a semi-log plot of FID amplitude versus time should be linear with a slope which gives the relaxation time T_2. For the oven-dried specimens, only a single exponential decay is present from which a relaxation time T_2 of 6 μs is obtained for both Kevlar and glass composites. This short decay time is indicative of tight molecular coupling and is representative of the decay times observed from solid materials.[6] For composites, this FID component is associated with the chemically-bound hydrogen atoms of the resin matrix for the case of the glass composite specimens and of both the matrix and the organic Kevlar fiber for the case of the Kevlar composite specimen.

Semi-log plots of the FID's for moisture conditioned specimens are shown in Figures 7 and 8 for Kevlar and glass composites, respectively. For both these specimens, the initial fast-decaying component with a time constant of 6 μs associated with tightly bound hydrogen is present as well as a moisture component beginning between 50 and 60 μs. The difference in time scales for the graphs in Figures 7 and 8 is due to the fact that the Kevlar moisture component was measurable at longer times than that for the glass composite due to the greater amount of moisture present in the Kevlar composite. As noted in these figures, the Kevlar composite decay curve exhibits an oscillation at the beginning of the moisture component whereas the glass specimens do not exhibit this feature (the Kevlar fiber NMR signals show this same oscillation as will be shown later in this section). Although it was not possible to investigate this feature within the scope of the program, it is tentatively interpreted as a "Lowe-Norberg beat" which is associated with hydrogen nuclei in different chemical environments having slightly different NMR frequencies caused by chemical exchange interactions.[7] Investigations of such features in the NMR signals could provide important information on the molecular structure changes accompanying moisture absorption in composites.

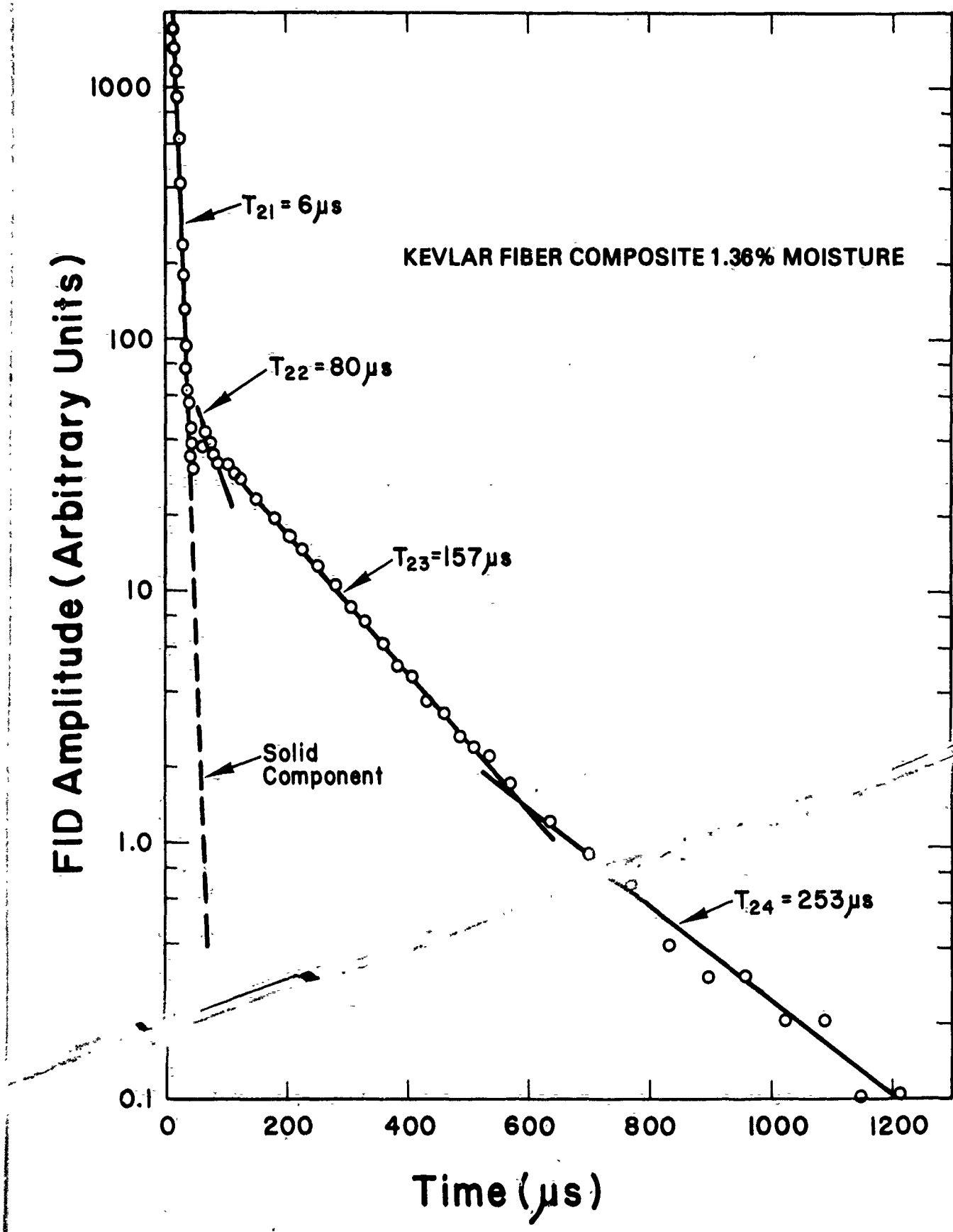


FIGURE 7. DETAILS OF FREE INDUCTION DECAY FOR KEVLAR COMPOSITE (MOISTURE = 1.36%)

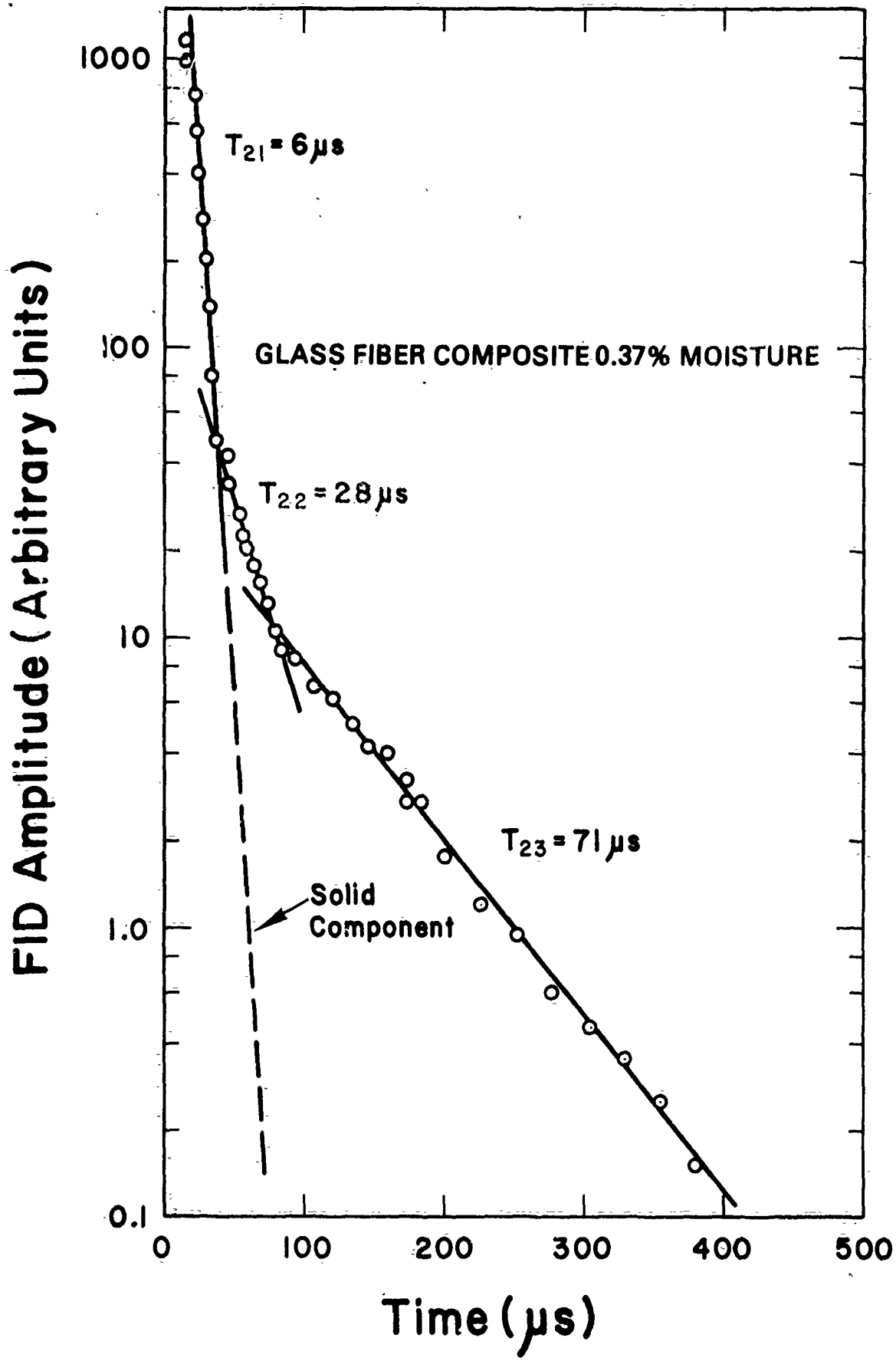


FIGURE 8. DETAILS OF FREE INDUCTION DECAY FOR GLASS COMPOSITE (MOISTURE = 0.37%)

For the Kevlar composite the moisture NMR signal is composed of three components with decay times as indicated in Figure 7 while for the glass composite only two moisture components are observed. These components may be associated with absorbed moisture in different states of molecular binding within the composites.

In Figures 9 and 10 are shown the absorbed moisture components of the FID's for the various moisture levels investigated for both the Kevlar and glass fiber composites. These plots represent averages of the NMR data obtained from all of the specimens measured (K2 - K5 and G2 - G5) at the various moisture levels investigated. For clarity, the initial part of the FID with the 6 μs time constant associated with the tightly bound hydrogen is not shown in these Figures and the data points are omitted; only the linear segments of the FID's beyond approximately 50 μs are presented. The lowest moisture level data shown in these figures (0.49% for Kevlar and 0.19% for glass) were obtained from the as-fabricated composite, that is, prior to oven drying and moisture conditioning. These results illustrate the sensitivity of the NMR method since even at these low moisture levels, NMR moisture signals were easily measured. Keeping in mind that the FID amplitude is plotted on a log scale, the NMR signal amplitude can be seen to vary strongly with moisture. This is shown more clearly in Figures 11 and 12 which show graphs of the FID signal amplitude at 200 μs versus percentage moisture. At low signal levels, the relationship is nonlinear due to non-linearities in the NMR spectrometer diode detectors. At higher signal levels a linear relationship is observed for the Kevlar composite.

Examination of Figures 9 and 10 show that in addition to the signal amplitude, the spin-spin relaxation times undergo a regular variation with moisture level. These results are tabulated in Table 3. As the moisture level increases, the time constants also increase indicating that additional moisture is less strongly absorbed in the composite system than is the case at low moisture levels. Also the distinction between the time constants for the separate moisture components becomes less pronounced as more moisture is absorbed into the composite systems.

In addition to the measurements on the composite specimens, a few NMR measurements were also performed on the S2 glass and Kevlar 49 fibers and the SP250/S2 glass and Reliabond 9350/Kevlar 49 prepregs. No proton NMR signals were observable from the S2 glass fiber. However, a strong NMR signal was observed from the Kevlar 49 fiber comparable in magnitude and characteristics to that observed from the Kevlar composite. An example of the free induction decay from less than one gram of Kevlar 49 fiber in the as-received condition is shown in Figure 13a. (Dry weight moisture percentage was determined by oven-drying the fiber after completion of the NMR experiments). As in the case of the composite, the Kevlar FID is characterized by a fast decaying initial part with a time constant of 6 μs (not readily seen in Figure 13a because of the time scale) and an absorbed moisture component beginning at approximately 60 μs. The oscillation observed in Figure 13a at the beginning of the moisture component for the Kevlar fiber implies that this feature in the NMR signals measured from the composite (see

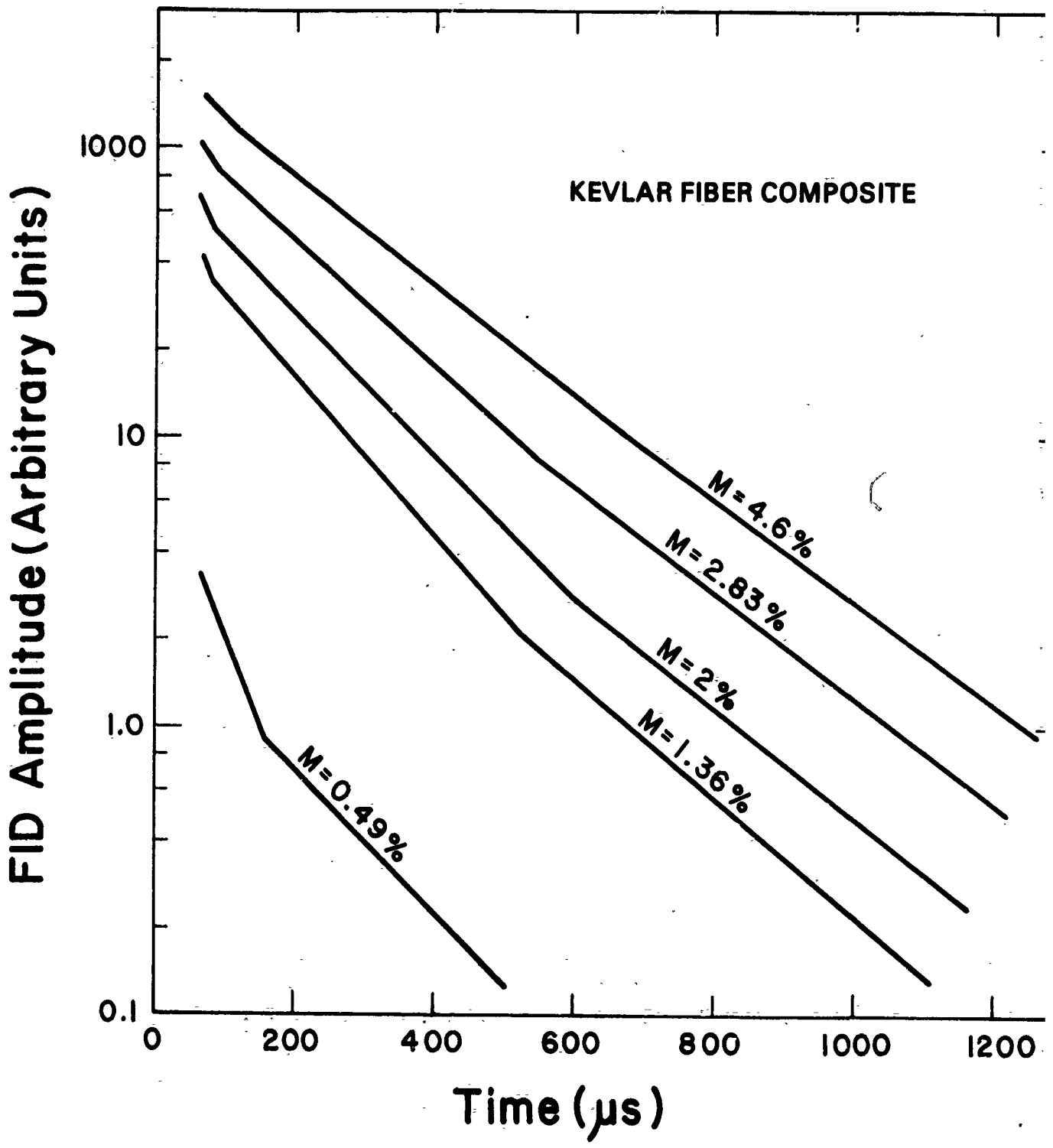


FIGURE 9. KEVLAR COMPOSITE FREE INDUCTION DECAYS FOR VARIOUS MOISTURE LEVELS

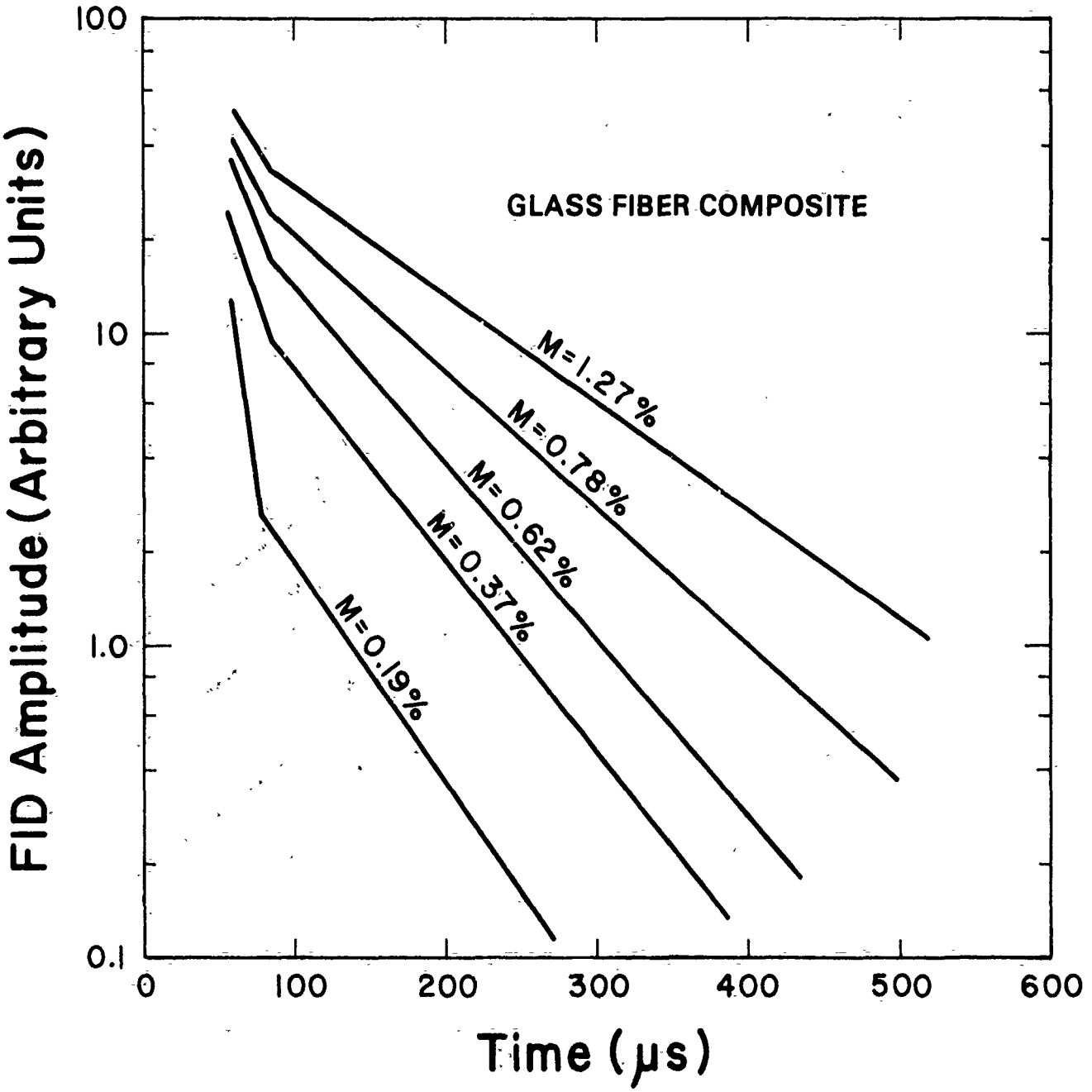


FIGURE 10. GLASS COMPOSITE FREE INDUCTION DECAYS FOR VARIOUS MOISTURE LEVELS

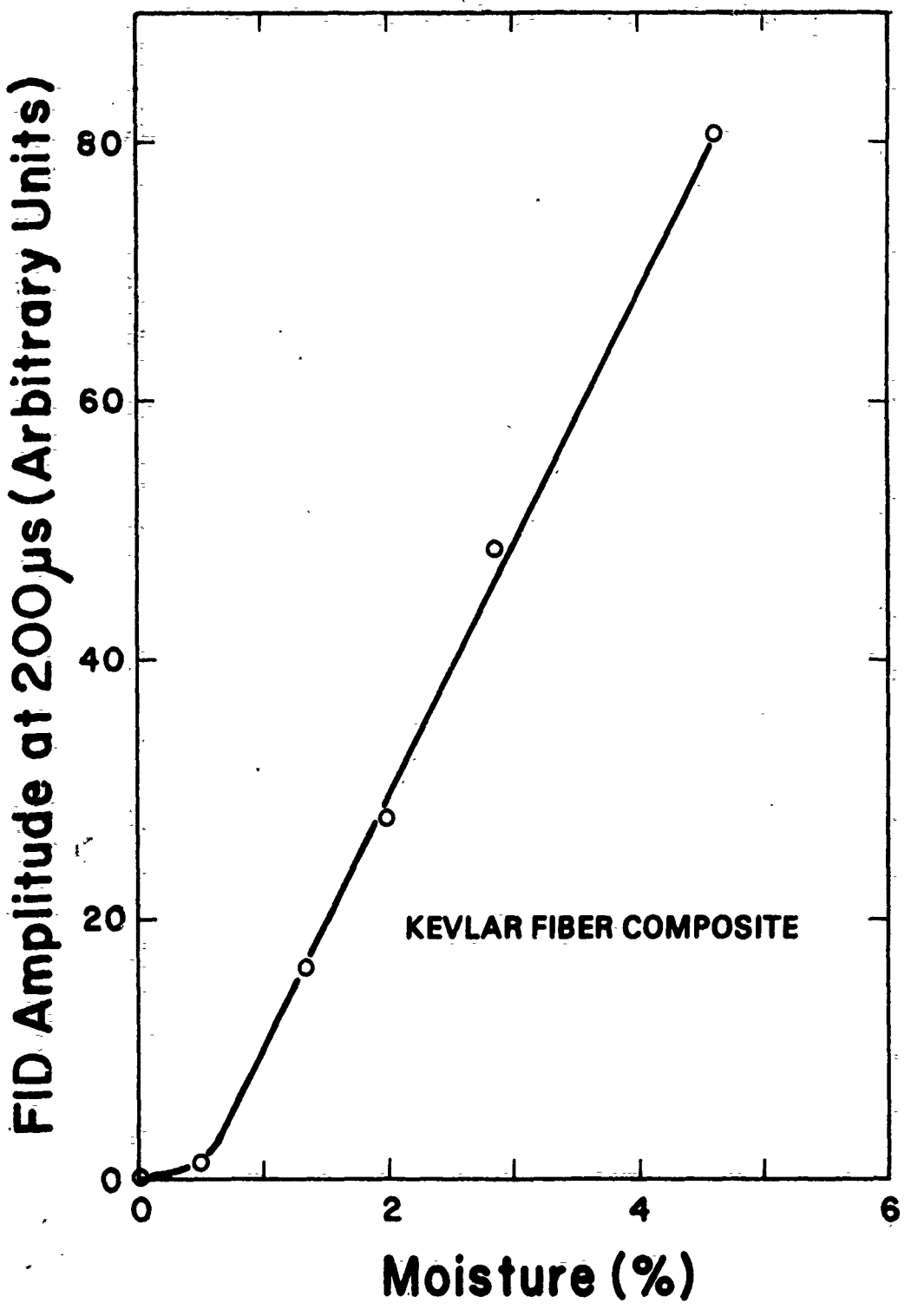


FIGURE 11. FREE INDUCTION DECAY VS MOISTURE FOR KEVLAR COMPOSITE

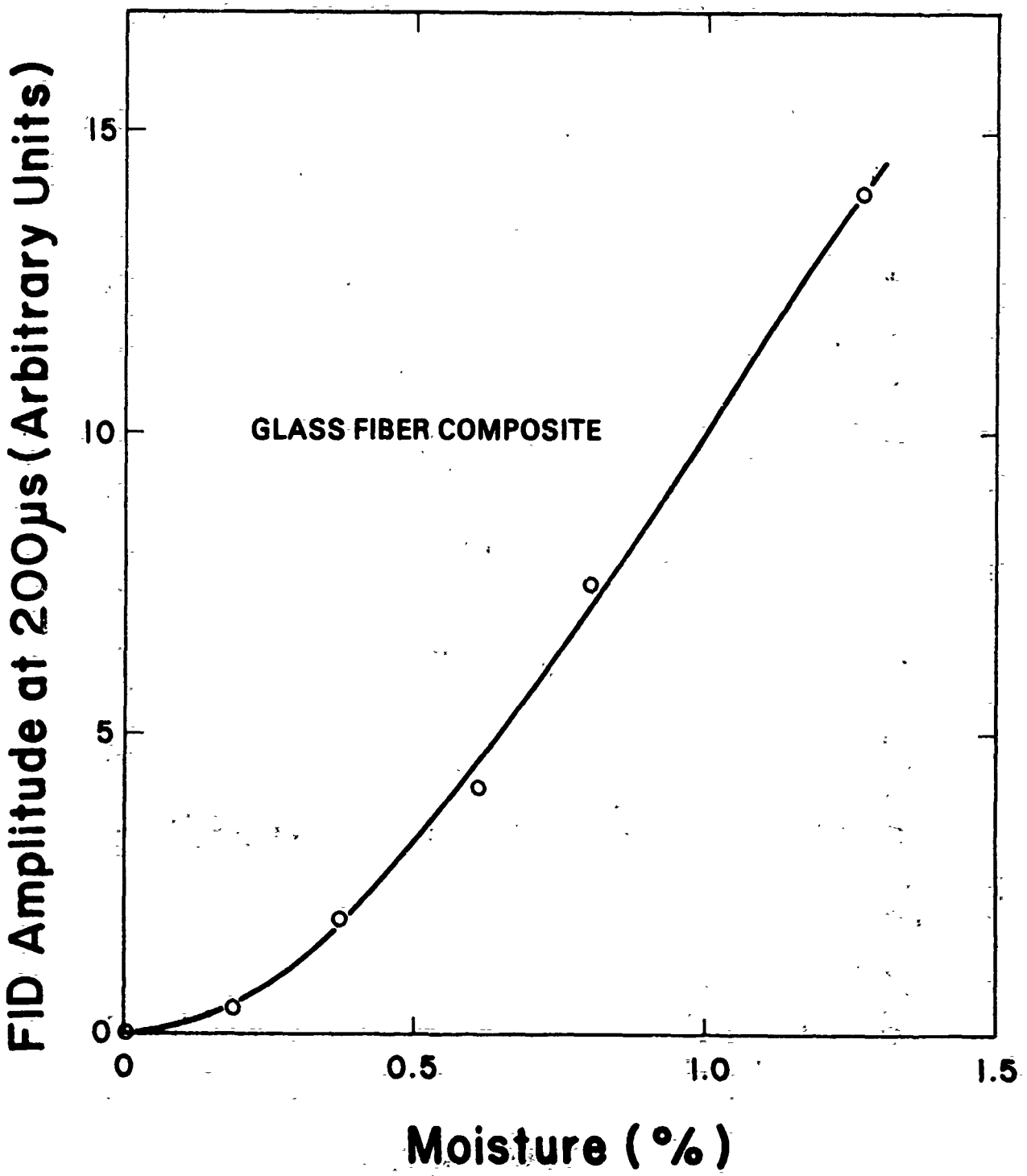
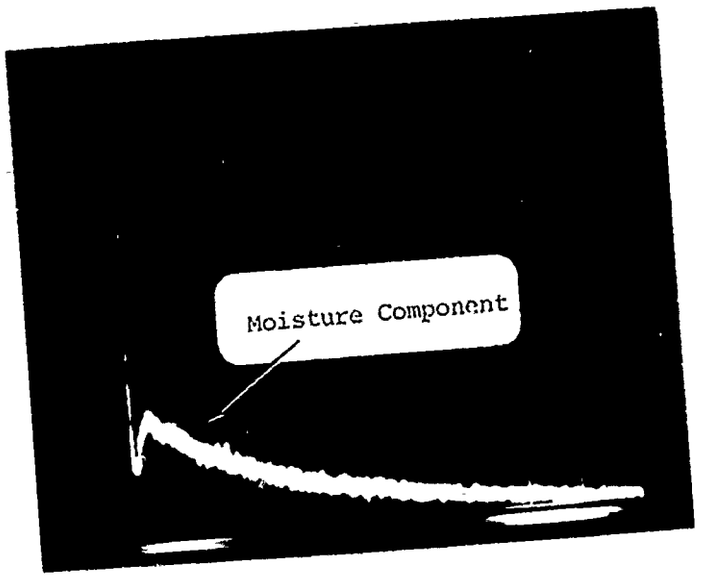


FIGURE 12. FREE INDUCTION DECAY VS MOISTURE FOR GLASS COMPOSITE

TABLE 3

SPIN-SPIN RELAXATION TIMES FOR MOISTURE IN KEVLAR AND GLASS COMPOSITE

Moisture (%)	Relaxation Times (μs) T_{22}	T_{23}	T_{24}
	Kevlar Composite		
0.49	70	175	---
1.36	75	157	210
2.00	82	175	228
2.83	113	200	235
4.60	178	230	245
	Glass Composite		
0.19	13	62	---
0.37	33	70	---
0.62	35	76	---
0.78	45	99	---
1.27	54	125	---

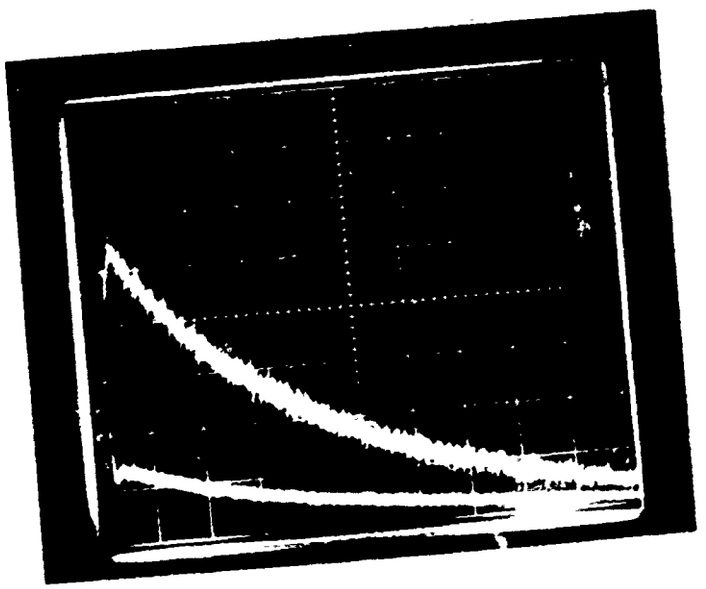


V - 0.2 V/div

H - 100 μs/div

Atten: - 16 DB

(a) Before Exposure to Environmental Chamber
(Weight = 0.988 g; moisture = 3%)



V (upper) - 0.05 V/div

V (lower) - 0.2 V/div

H - 200 μs/div

Atten: - 22 DB

(b) After Weight Gain of 0.11 g
(moisture - 14.0%)

FIGURE 14. FREE INDUCTION DECAYS FROM KEVLAR 49 FIBER

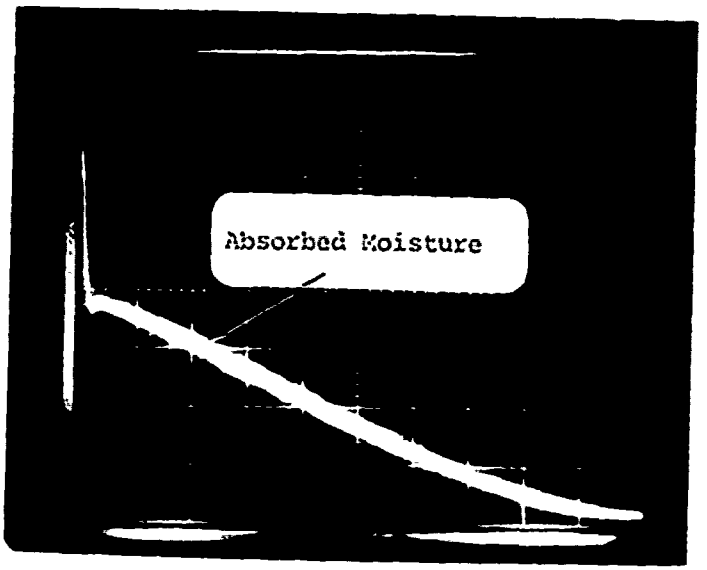
Figure 3b) is associated with the fibers in the Kevlar composites.

NMR measurements were also made on the Kevlar fiber after exposure to the same environmental conditions as the composite specimens, namely 51.6°C (125°F) and 95% relative humidity. Results for the Kevlar fiber after an environmental exposure of 29 hours and a weight gain of 0.11 grams are shown in Figure 13b. The large increase in the FID moisture component due to the absorbed moisture is readily seen (note the change in attenuator setting from Figure 13a. Experimental difficulties were encountered in performing the NMR measurements on the fiber since the weight was found to change quite rapidly once the fiber was removed from the environmental chamber. In fact the NMR signal could be observed diminishing in amplitude with time while the fiber specimen was outside the environmental chamber.

Results of NMR measurements made on the Kevlar fiber after long term (approximately 3 months) exposure in the environmental chamber are shown in Figure 14. A very strong absorbed moisture FID component was observed as shown in Figure 14a after a total weight gain of more than 1 gram which is an increase of over 100% from the original weight. In addition to the large absorbed moisture component, another distinct FID component was observed at a longer time as seen in the expanded scale photograph shown in Figure 14b. This signal component has the time constant characteristics of free water and can probably be associated with the free moisture adhering to the fiber surface (the fiber was wrapped on a teflon bobbin for the NMR measurements so that excess moisture within the fiber windings could not be readily removed). The NMR signal obtained after allowing the Kevlar fiber to dry overnight at ambient conditions is shown in Figure 15a. The absorbed moisture signal is seen to have diminished greatly and the free moisture signal is no longer present. After oven-drying the fiber, no moisture signal component was observed as illustrated in Figure 15b.

The Kevlar and glass prepregs were kept frozen at -16.7°C (2°F) until ready for measurement. Upon removal from the freezer and thawing at room temperature, the free induction decays shown in Figure 16 were obtained. The prepregs were exposed to ambient laboratory conditions for a period of one month at which time the weight of the glass prepreg was found to be virtually unchanged whereas the Kevlar prepreg had increased in weight approximately 0.4%. No change in the NMR signals from the prepregs after the one month exposure to ambient conditions could be seen compared with the signals obtained from the prepregs after initial removal from the freezer.

Interesting NMR results were obtained from one of the fractured moisture conditioned glass composite specimens after destructive testing. FID's obtained from the fractured specimen are shown in Figure 17. A strong absorbed moisture component is observed as seen in Figures 17a and 17b. It is probably due to moisture that was absorbed through the fracture surfaces (the fractured specimen pieces were placed back in the environmental chamber after destructive testing). Figure 17c is an expanded scale photograph showing the presence of a free moisture signal at approximately the 2 ms point. Since the specimen surface was wiped dry of excess moisture, the free moisture NMR signal is no doubt due to moisture which entered the interior of the composite through fracture planes.



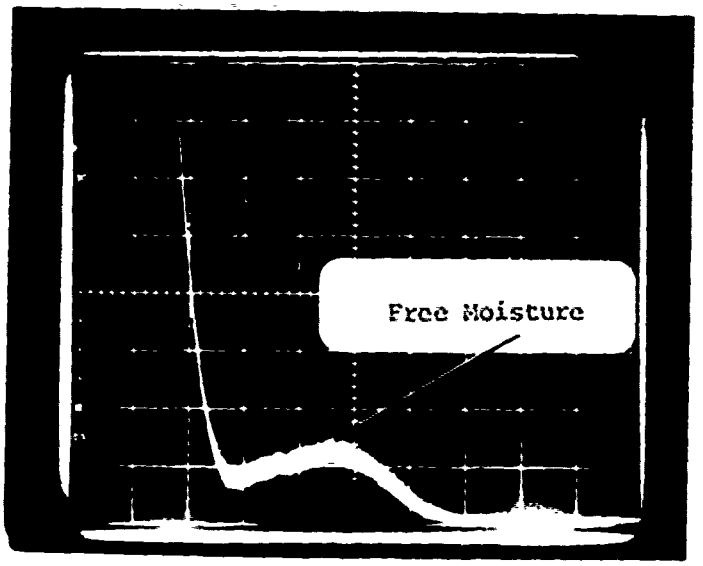
V - 0.2 V/div

H - 200 μs/div

Atten: - 28 DB

(a) After Weight Gain of 1.095 g (Moisture = 117%)

5925



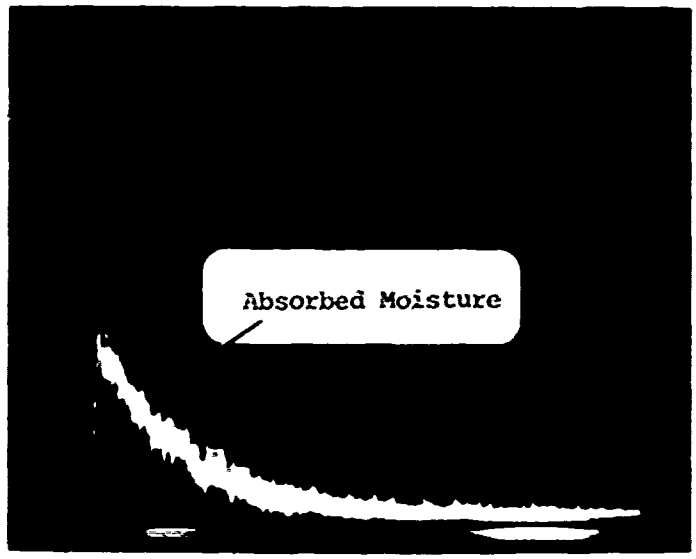
V - 0.02 V/div

H - 1000 μs/div

Atten: - 28 DB

(b) Same as (a) but Expanded Scales

FIGURE 14. EFFECTS OF ENVIRONMENTAL EXPOSURE ON FREE INDUCTION DECAYS FROM KEVLAR 49 FIBER

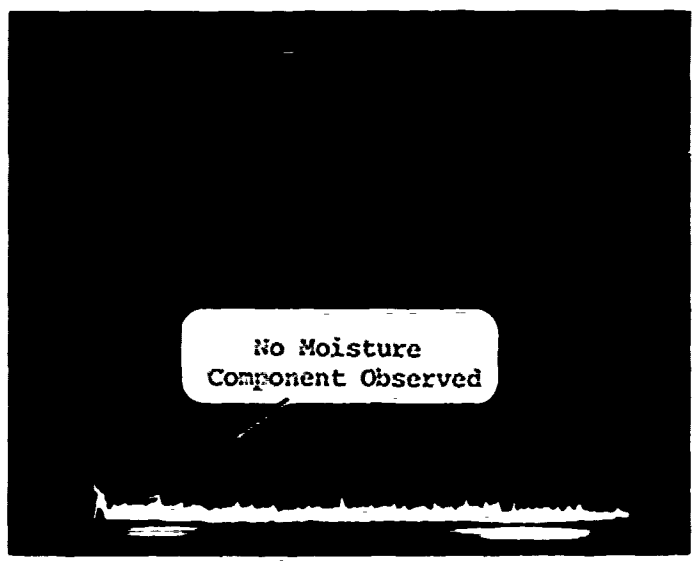


V - 0.02 V/div

H - 200 μs/div

Atten: - 28 DB

(a) After Overnight Room Drying
(moisture = 4.6%)



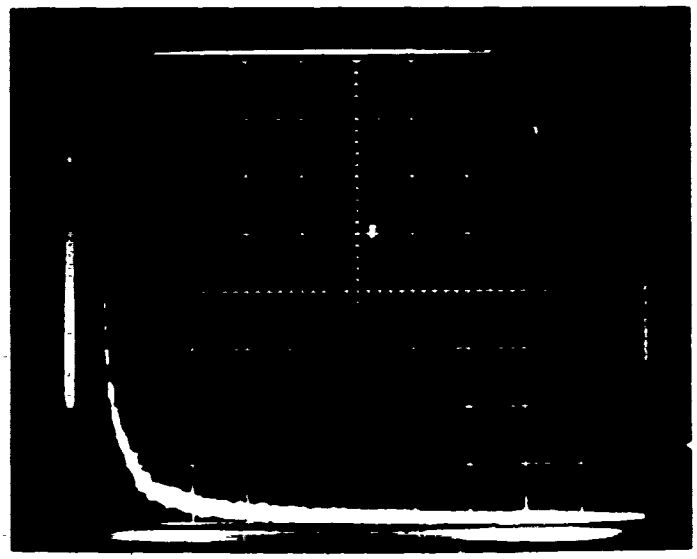
V - 0.02 V/div

H - 100 μs/div

Atten: - 28 DB

(b) After Oven Drying

FIGURE 15. FREE INDUCTION DECAYS FROM DRIED KEVLAR 49 FIBER

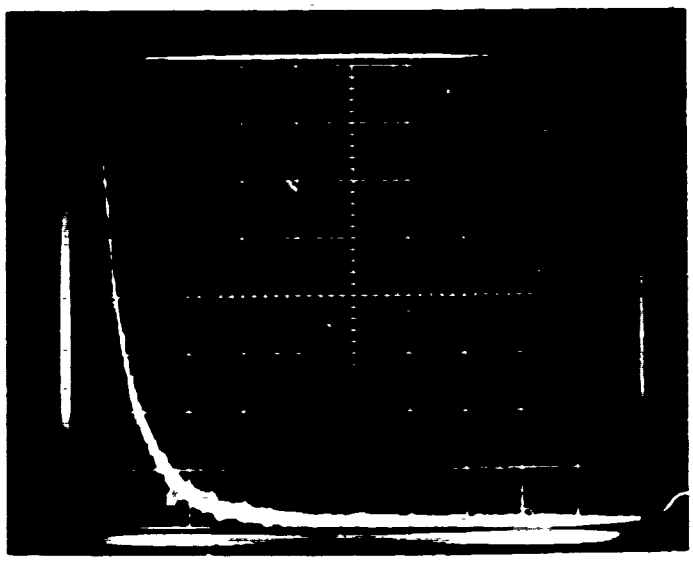


V - 0.02 V/div

H - 100 μs/div

Atten: - 28 DB

(a) [illegible]



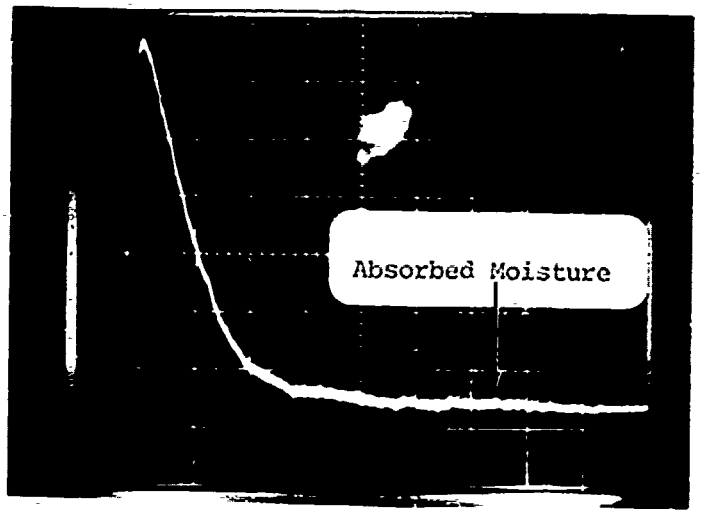
V - 0.02 V/div

H - 100 μs/div

Atten: - 28 DB

(b) [illegible]

[illegible] COMPOSITE PREPREGS

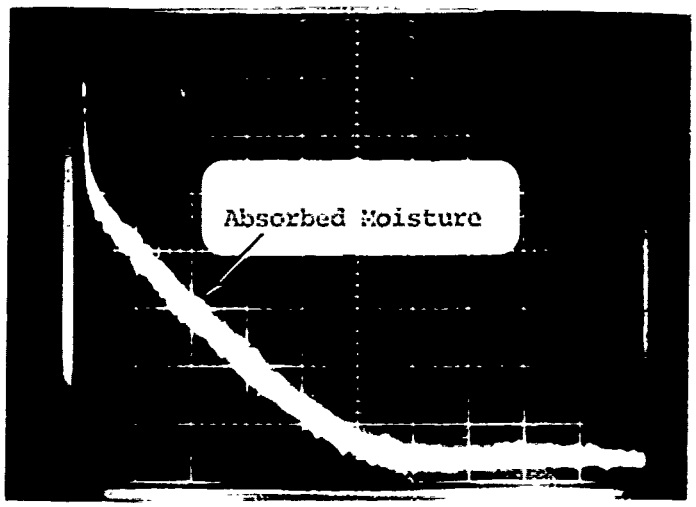


(a)

V - 0.2 V/div

H - 10 μs/div

Atten: - 23 DB



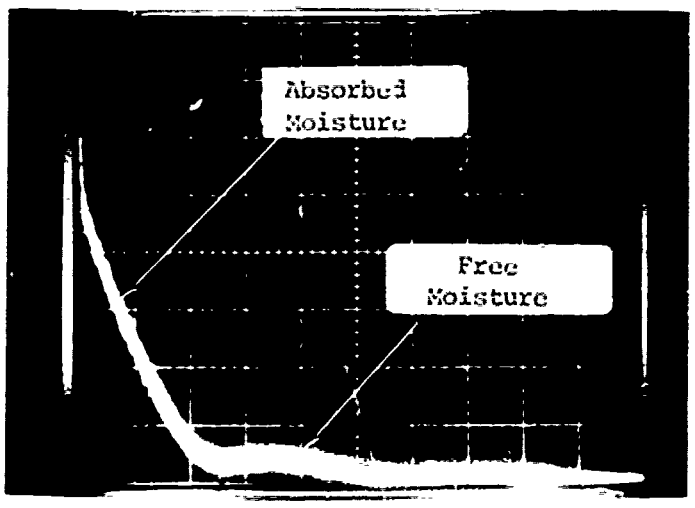
(b)

V - 0.05 V/div

H - 200 μs/div

Atten: - 23 DB

5028



(c)

V - 0.05 V/div

H - 500 μs/div

Atten: - 23 DB

FIGURE 17. FREE INDUCTION DECAYS FROM FRACTURED GLASS FIBER COMPOSITE

Mechanical Testing

Tabulations of the tensile test results for the dry and moisture saturated composite specimens are presented in Tables 4 and 5. As indicated in the tables, some of the specimens failed outside the 5.1 cm (2 in.) gage length of the extensometer, however, none of the specimens failed at the grips. Moisture saturation was found to reduce the average ultimate tensile strength 4.3% for the glass composite and 14% for the Kevlar composite while the tangent modulus increased 11.6% for the glass composite and 10.9% for the Kevlar composite.

Stress-strain curves for the Kevlar and glass composites are shown in Figures 18 and 19. The curves in these figures are the best-fit polynominal curves (using 3 degrees of regression) to all of the recorded stress-strain curves and thus represent the best overall average curves for the specimens tested. The stress-strain curves for the dry and moisture saturated specimens are included together in the figures for comparison. The changes in stress-strain characteristics produced by environmental exposure can be readily seen from these figures.

TABLE 4

SUMMARY OF TENSILE TEST DATA FOR DRY GLASS AND DRY KEVLAR COMPOSITES

Head Displacement Rate: 0.05 in./sec

Specimen No.*	Width (in.)	Thickness (in.)	Area ($in.^2$)	Ultimate Tensile Strength (kpsi)	Tangent Modulus (Mpsi)	Maximum Strain (%)
GLASS COMPOSITE						
G22	1.010	0.073	0.07373	26.4	1.98	***
G21	1.009	0.074	0.07466**	25.2	1.81	11.86
G14	1.009	0.073	0.07365**	26.4	1.69	11.55
G20	1.003	0.073	0.73200	27.0	1.69	12.30
G18	0.992	0.072	0.07142	26.2	1.75	11.72
G17	1.038	0.072	0.07474	26.8	1.73	11.76
G16	1.014	0.072	0.07301	26.6	1.69	12.58
G15	1.014	0.072	0.07301**	27.1	1.67	12.78
G13	1.014	0.073	0.07402	26.0	1.63	11.43
G19	1.024	0.074	0.07578**	26.3	1.58	12.74
			Avg.	26.4	1.72	12.08
KEVLAR COMPOSITE						
K19	1.013	0.090	0.0912	28.4	0.798	8.31
K17	0.998	0.091	0.0908**	29.8	0.920	7.95
K20	1.024	0.090	0.0922	28.0	0.877	7.40
K15	1.008	0.090	0.0907	29.2	0.914	7.02
K22	1.022	0.090	0.0920**	27.2	0.840	10.48
K21	1.005	0.092	0.0925**	26.8	0.863	9.47
K13	1.013	0.091	0.0922**	27.3	0.913	8.04
K14	1.015	0.092	0.0934**	28.0	0.866	7.42
K18	1.014	0.090	0.0913	29.7	0.942	8.12
K16	1.012	0.090	0.0911	30.9	0.912	8.00
			Avg.	28.5	0.884	8.22

1 in. = 2.54 cm.
1 psi = 6.9 kPa

* Specimens listed in chronological order of testing.
** Specimens failed within the 2-in. gage length; all others failed outside the gage length.
*** No data.

TABLE 5

SUMMARY OF TENSILE TEST DATA FOR GLASS AND KEVLAR COMPOSITE AFTER SATURATION AT 51.6°C (125°F) AND 95% RELATIVE HUMIDITY

Head Displacement Rate: 0.05 in./sec.

GLASS COMPOSITE

Specimen No.*	Width (in.)	Thickness (in.)	Area ($in.^2$)	Ultimate Tensile Strength (kpsi)	Tangent Modulus (Mpsi)	Maximum Strain (%)
G2	1.013	0.073	0.0739	23.3**	1.91	11.78
G3	1.012	0.073	0.0739	23.5	1.98	11.71
G4	1.012	0.073	0.0739	23.4	1.98	11.87
G5	1.008	0.072	0.0726	23.0	1.95	10.97
G6	1.013	0.072	0.0729	22.9**	1.94	11.37
G7	1.012	0.073	0.0739	23.0**	1.82	12.15
G8	1.014	0.074	0.0750	23.0**	2.28	12.24
G9	1.013	0.075	0.0760	23.2**	1.86	12.46
G10	1.013	0.075	0.0760	22.8	1.77	11.34
G11	1.015	0.075	0.0761	23.3	1.73	11.23
			Avg.	23.1	1.92	11.71

KEVLAR COMPOSITE

K2	0.995	0.092	0.0915	24.1**	0.98	9.09
K3	1.005	0.097	0.0975	22.6	0.94	8.33
K4	1.013	0.094	0.0952	23.4	0.98	7.06
K5	1.012	0.094	0.0951	23.8	0.96	7.23
K6	1.010	0.093	0.0939	23.9	0.99	6.77
K7	1.012	0.094	0.0951	24.1	1.01	6.60
K8	1.012	0.092	0.0931	26.7	1.04	7.85
K9	1.002	0.095	0.0952	***	***	***
K10	1.015	0.090	0.0916	26.7	0.97	11.33
K11	1.013	0.089	0.0902	25.2	0.96	9.35
			Avg.	24.5	0.98	8.18

1 in. = 2.54 cm
1 psi = 6.9 kPa

* Specimens listed in chronological order of testing.
** Specimens failed within the 2-in. gage length, all others failed.
*** No data.

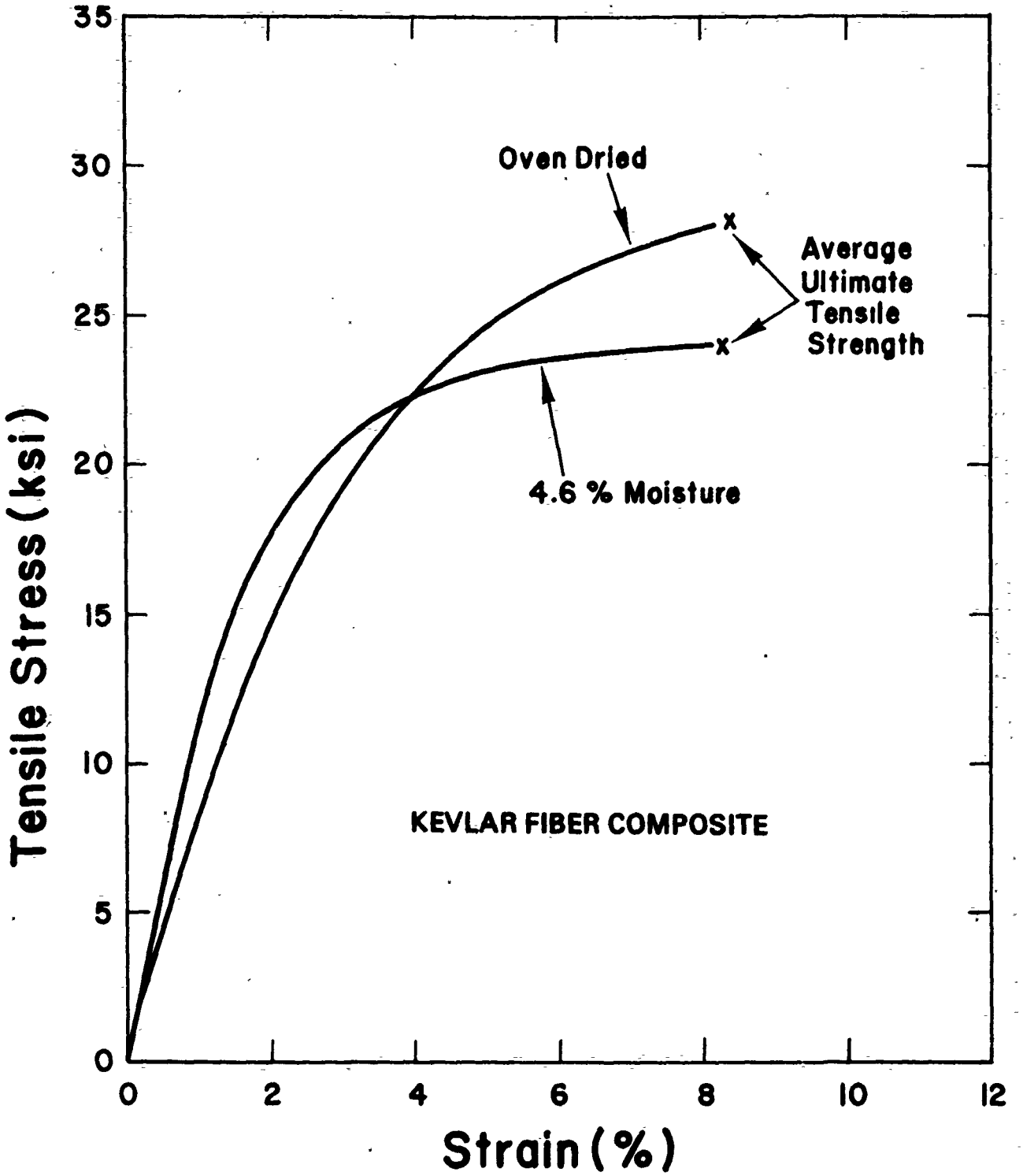


FIGURE 18. STRESS-STRAIN CURVES FOR KEVLAR COMPOSITE

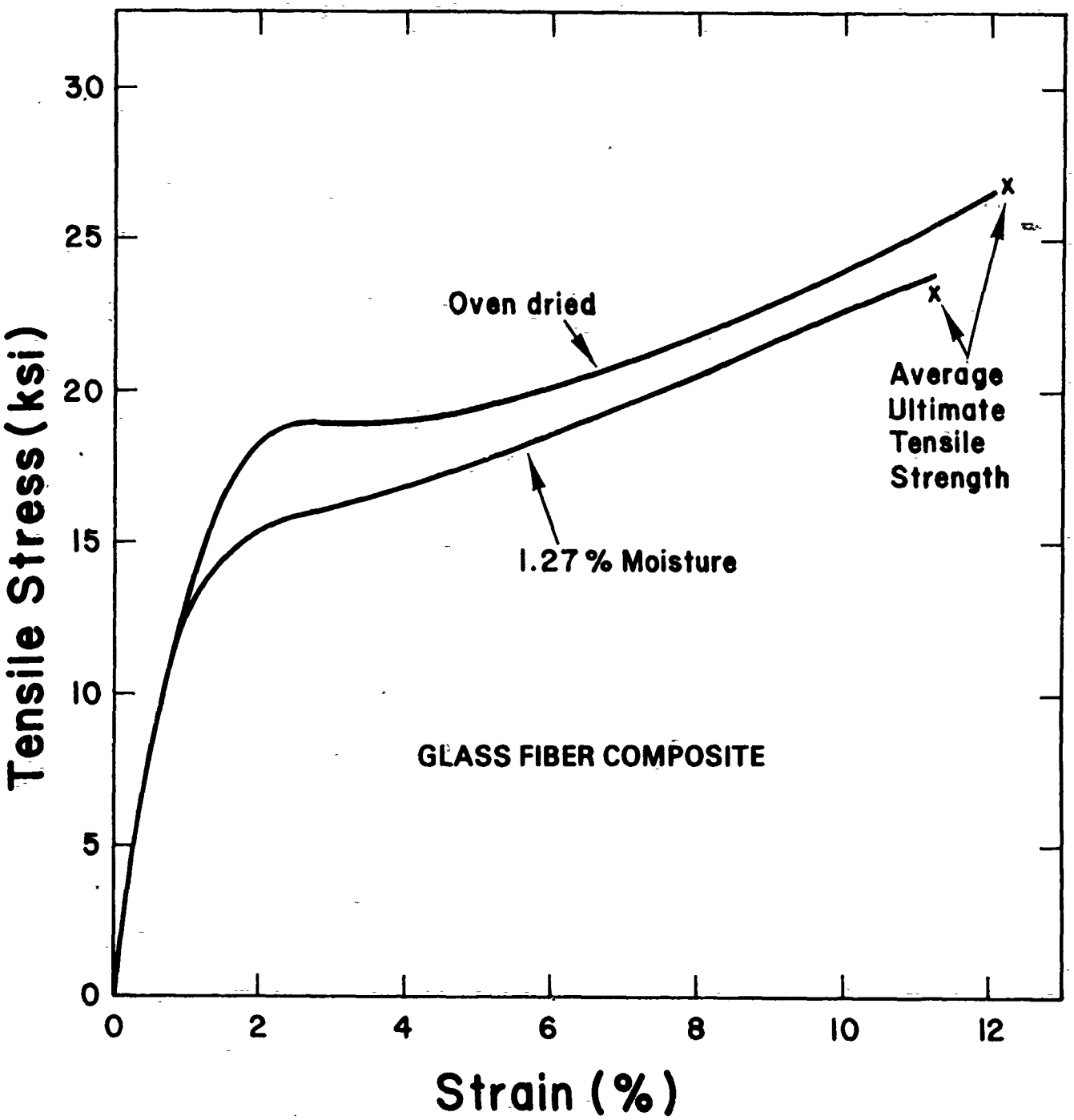


FIGURE 19. STRESS-STRAIN CURVES FOR GLASS COMPOSITE

IV. DISCUSSION

The results of this project are very encouraging in demonstrating the feasibility of NMR to not only nondestructively measure moisture in organic matrix composites but also to serve as a tool in laboratory studies of the relationship between moisture absorption and mechanical degradation. Although the ultimate sensitivity of the technique for moisture detection has not been determined, the results obtained in this project show that moisture levels of 0.2% can be readily measured with NMR and variations in moisture on the order of 0.1% can be resolved. The existence of multiple components for the absorbed moisture free induction decay signals indicates that the absorbed moisture exists in several states of molecular binding. Similar results have been obtained for various other heterogeneous materials such as soil, concrete, paper and food products.[8] In the case of the Kevlar composite, moisture is absorbed by the organic fiber as well as by the matrix, while in the case of the glass composite, moisture is absorbed by the matrix alone driven by environmental temperature and humidity gradients.

It is known that moisture can be transported through the matrix by several different mechanisms such as:[9]

1. Capillary action along the fiber-matrix interface,
2. Voids, pores, or cracks in the resin system,
3. Diffusion through the resin itself.

It is plausible therefore, that the distinct components observed in the absorbed moisture FID's may associated with water in these various states. The fastest-decaying absorbed moisture component, that is, T_{22}, can be associated with the moisture absorbed along the fiber-matrix interface since these moisture molecules are expected to be relatively tightly bound by surface tension and such conditions produce a fast decay; the longer time constant FID components can be associated with moisture absorbed into the resin. The additional FID component observed for the Kevlar composite compared with the glass composite may be due to the fact that the Kevlar fiber itself absorbs considerable moisture. More detailed investigations of the Kevlar fiber NMR characteristics and those of neat resins would be needed in order to verify these interpretations.

The tensile test results illustrate the adverse effect environmental exposure has on the mechanical properties of composites, particularly strength. Although the exact mechanisms of mechanical degradation are not clearly understood, two mechanisms which can result in a loss of strength are "plasticizing" of the matrix caused by moisture absorption and microcracking of the composite structure due to swelling of the matrix.[10] Moisture existing in microcracks would be expected to be relatively free with a long T_2 time constant. However, there was no evidence of a long time constant, free moisture NMR signal component in the measurements made on the environmentally conditioned specimens prior to mechanical testing. (Although a strong free

moisture signal was observed from the fractured specimens which had been placed back in the environmental chamber as shown in Figure 15). If free moisture were present in microcracks, it should have been detected by NMR. Hence, the most likely cause of the measured mechanical degradation is plasticizing of the matrix. This conclusion is further substantiated by the general increase in relaxation times observed with increasing moisture absorption. The longer relaxation times imply looser molecular coupling which could accompany plasticizing of the matrix resin.

As a result of the work reported here, several areas are suggested for utilizing NMR in further exploring and understanding the relationship between moisture absorption and mechanical degradation. Most work reported on composites has involved equilibrium moisture concentrations where there is no moisture gradient through the thickness of the specimen. In practical situations, however, the distribution will not be uniform due to surface absorption and desorption. This fact could cause discrepancies in test data because some plys will be more moisture-saturated than others and therefore will be more prone to loss in strength than neighboring plys which are less saturated. By utilizing inhomogeneous magnetic fields and field-focusing techniques,[11] it may be feasible to utilize NMR to nondestructively determine the moisture concentration distribution in a composite. Also in practical situations, composite structures in various environmental atmospheres will often be simultaneously subjected to loads. Uncertainty presently exists on whether the level of strain affects the moisture absorption of composites. Since it is known that an applied stress can influence the flow of water in a porous medium, there is a question of whether or not the level of stress will influence diffusion of moisture in composites. In one report, it is shown that for isotropic bodies the elastostatic problem can be uncoupled from the diffusion relation and therefore the level of strain should not affect the concentration of moisture.[12] However, in another report, data are presented which show that loaded specimens gain considerably more weight in a humidity test than unstrained specimens.[13] Since this work was performed at relatively high levels of applied strain, there could have been considerable cracking and void formation within the specimen resulting in high weight gain values. Since NMR is capable of distinguishing between free moisture entering through cracks and absorbed moisture diffusing through the matrix, it could provide important information on the question of the influence of applied stress on moisture absorption.

Although further laboratory studies are needed to better understand the relationship between the various components of NMR signals and moisture absorption, the results of this program indicate that NMR could be utilized for practical measurement of moisture content. Based on available technology, large cavity NMR instrumentation systems could be developed for moisture measurement in certain types of components, such as helicopter rotorblades. For measurements on larger structures, such as aircraft components, the technology exists for developing an NMR system capable of making measurements from one surface of a large specimen. This approach could also be applied to large composite panels for quality control during fabrication.

V. CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1. The feasibility of applying pulsed NMR to the study of moisture absorption, mechanical degradation and other effects in structural organic matrix composites was demonstrated.

2. NMR is a sensitive technique for nondestructively measuring the amount of moisture present in organic matrix composites due to environmental exposure. Under the conditions utilized for the experiments reported, moisture levels of 0.2% could be detected and variations in moisture of less than 0.1% could be resolved.

3. Multi-component NMR signals were observed which provide information on hydrogen atoms in various states of binding in the composites. The absorbed moisture NMR signal components are readily distinguishable from the signal component arising from the chemically bound structural hydrogen atoms. The distinct components of the absorbed moisture NMR signal may be associated with moisture absorbed along the fiber-matrix interface and moisture absorbed into the matrix resin. Additional study of the various features of the NMR signals could potentially provide important information on the mechanisms of moisture absorption in composites.

4. Results of tensile testing showed that moisture absorption reduced the tensile strength by 4.3% for the glass composite and by 14% for the Kevlar composite. Analysis of the NMR signals indicates that matrix plasticizing is more likely the cause of mechanical degradation in the composites than moisture entering through cracks and voids.

5. Measurements on fractured composite specimens showed that NMR could be utilized to measure the amount of free water entering a composite through cracks and fissures.

Recommendations

1. Perform measurements at low moisture levels to determine the sensitivity and resolution of the NMR method for moisture detection.

2. Perform detailed NMR and moisture conditioning experiments on the component materials such as fibers, resin, and prepregs in conjunction with the complete composite to obtain information on the composition of the observed NMR signals relative to the component materials.

3. Measure the spin lattice relaxation time, T_1, as a function of moisture absorption and mechanical degradation in order to obtain a more complete understanding of the molecular mechanisms involved in mechanical degradation due to environmental exposure.

4. Utilize computational methods to separate the T_1 and T_2 data into individual components and to determine the percentage contribution of each component to the total signal. This analysis would provide quantitative information on the relative changes in NMR signal components associated with moisture in different states of molecular binding.

5. Investigate the effect of applied stress on moisture absorption utilizing NMR to determine the presence of free water entering the specimens through cracks and the amount of moisture diffusion in the matrix.

6. Explore the feasibility of utilizing inhomogeneous magnetic fields and field-focusing techniques to measure moisture gradients and moisture profiles with NMR.

7. Perform the developmental work necessary for utilizing NMR as a practical nondestructive test method for measuring the amount of moisture present in composites.

VI. LIST OF REFERENCES

1. Advanced Composite Materials-Environmental Effects, ASTM STP 658, Ed. by J. R. Vinson, American Society for Testing and Materials, Philadelphia, 1978.

2. Rollwitz, W. L., "Moisture Measurements in Various Hygroscopic Materials Using Nuclear Magnetic Resonance," in Humidity and Moisture Measurement and Control in Science and Industry, Vol. 2, Ed. by Wexler, A., Reinhold Publ. Co., 1965.

3. Rollwitz, W. L., "On Stream NMR Process Control," J. of the American Oil Chemists Soc., 48, No. 2, pp. 59-66, 1971.

4. Matzkanin, G. A., and Gardner, C. G., "Nuclear Magnetic Resonance Sensors for Moisture Measurement in Roadways," Transportation Research Record 532, pp. 77-86, 1975.

5. Shen, C., and Springer, G. S., J. of Composite Materials, 10, pp. 2-20, 1976.

6. Abragam, A., The Principles of Nuclear Magnetism, Clarendon Press, Oxford 1961.

7. Lowe, I. J., and Norberg, R. E., "Free Induction Decays in Solids," Phys, Rev., 107, No. 1, pp. 46-61, 1957.

8. Rollwitz, W. L., "Transient NMR Measurements," J. of the American Oil Chemists Soc., 48, No. 2, pp. 67-69, 1971.

9. Cook, T. S., Walrath, D. E., and Francis, P. H., "Moisture Absorption in Graphite-Epoxy Composite Materials," Proc. 22nd National SAMPE Symposium and Exhibition, 22, pp. 339-349, 1977.

10. Miller, A. K. and Adams, D. F., "Inelastic Micromechanical Analysis of Graphite/Epoxy Composites Subjected to Hygrothermal Cycling," Advanced Composite Materials-Environmental Effects, ASTM STP 658, Ed. by J. R. Vinson, American Society for Testing and Materials, Philadelphia, pp. 121-142, 1978.

11. Sundheim, B. R., "Diffraction and Microscopy in Solids and Liquids by NMR," European Scientific Notes-London, p. 297, 1975.

12. Gurtin, M. E., presented at "Mechanics of Composites Review," Dayton, Ohio, 26-28, October 1976.

13. Broutman, L. J., presented at Mechanics of Composites Review," Dayton, Ohio, 26-28, October, 1976.

APPENDIX A

THE EFFECT OF MOISTURE ON THE MECHANICAL PROPERTIES OF ORGANIC MATRIX COMPOSITE AND NONDESTRUCTIVE DETECTION OF MOISTURE - A BRIEF SUMMARY OF THE STATE-OF-THE-ART

During performance of the project which is the subject of this report, a literature search was conducted on the effect of moisture on the mechanical properties of organic matrix composites and methods for nondestructively detecting and measuring moisture in composites. The intention in performing this literature search was to obtain information on the current state-of-the-art regarding environmental mechanical degradation of composites. Because of the large amount of information revealed by the literature search, it was decided to perform a more extensive review of the literature in this area and make the results available to the public in the form of a Nondestructive Testing Information Analysis Center (NTIAC) publication. This publication will be forthcoming in the near future. Presented below is a brief summary of the state-of-the-art based on a review of some ofthe documents identified during the literature search.

LITERATURE SEARCH STRATEGY

The computer retrieval facilities of the Nondestructive Testing Analysis Center (NTIAC) located at Southwest Research Institute were used in compiling a bibliography of source documents for the state-of-the-art survey. This search covered technical journals, conference proceedings, research reports, and department of defense technical reports from 1960 through 1979. Additional manual searching was performed to obtain information on more recently reported work in the areas of interest.

The general computer search strategy is illustrated in Table A-1. In some of the computer files searched, the strategy illustrated in Table A-1 had to be modified slightly with respect to terminology. In the search, one term from each group of terms separated by an "and" must be associated with the document for it to appear as a find. Group A contains terminology relevant to composite materials; Group B includes terminology related to NMR and moisture; and Group C contains terminology related to testing or inspection and mechanical properties. The "?" after some terms means that the term is truncated, that is, all terms having the same initial letters in the same order will be searched. Thus, "MEASUR?" will search for measure, measures, measured, measuring, etc.

The specific documentation files searched and the results are shown in Table A-2. In addition to the NTIAC and DDC files, the open literature searches utilized the LOCKHEED/DIALOG System to search the following files:

(1) COMPENDEX (COMPuterized ENgineering inDEX),
(2) National Technical Information Service (NTIS),
(3) ISMEC-Mechanical Engineering,
(4) Chemical Abstracts.

TABLE A-1

LITERATURE SEARCH STRATEGY

GROUP A		GROUP B		GROUP C
COMPOSITE(W)MATERIAL?		NMR		MEASUR?
COMPOSITE(W)STRUCTURE?		NUCLEAR(W)MAGNETIC(W)RESONANCE		DETECT?
FIBER(W)REINFORCED		NUCLEAR(W)RESONANCE		DETERMINATION
MATRIX(W)MATERIAL?	and	MAGNETIC(W)RESONANCE	and	INSTRUMENTATION
CARBON(W)REINFORCED		MOISTURE		NONDESTRUCT?
CARBON(W)FIBER		MOISTURE(W)CONTENT		DEGRADATION
GRAPHITE(W)FIBER		WATER		MECHANICAL(W)PROPERTIES
GRAPHITE(W)REINFORCED		HUMIDITY		BULK(W)MODULUS
GRAPHITE(W)COMPOSITE?				ELASTIC(W)PROPERTIES
GLASS(W)FIBER?				FATIGUE
GALASS(W)REINFORCED				STRENGTH
GLASS(W)REINFORCED				TENSILE(W)PROPERTIES
FIBERGLASS				TOUGHNESS
REINFORCED(W)PLASTIC?				
KEVLAR?				

As illustrated in Table A-2, the total number of literature finds resulting from these searches was 1133. Review of the bibliographies of these searches indicated that approximately 150 documents were relevant to the specific areas of interest, namely, the effect of moisture on the mechanical properties of organic matrix composites and the nondestructive detection of moisture in composites. The state-of-the-art in these two areas based on a partial review of the documents resulting from the literature search will be briefly described in the following paragraphs.

MECHANICAL PROPERTY DEGRADATION

Most of the studies directed toward determining the effect of moisture on the mechanical properties of composites have concluded that absorbed moisture affects primarily the matrix- and interface-dominated properties of the composite and leads to significant degradation and changes in failure modes.[1-4] Filament-dominated properties show very little environmental sensitivity.[4,5] According to Deiasi and Whiteside,[6] epoxy resins absorb atmospheric moisture by instantaneous surface absorption and subsequent diffusion through the interior. The absorbed water is not liquid but exists rather in the form of hydrogen-bonded molecules or clusters within the polymer. Liquid water may, however, be transported by capillary action along cracks and along fiber-matrix interfaces and may appear at interior voids. The absorbed water softens epoxy resins, causes them to swell and lowers their glass transition temperature. A number of investigations have shown the moisture diffusion in resins and composites to be described by Fick's Law and the temperature dependence of moisture diffusivity to obey an Arrhenius relationship.[7]

The mechanisms and effect of moisture ingress into advanced composite materials are presented in a recent ASTM Special Technical Publication entitled Advanced Composite Materials-Environmental Effects.[8] Results are presented of a number of studies on absorption and diffusion, thermal cycling, application of external stress, and the hygrothermal mechanics of moisture diffusion. It was found, for example, that Fick's Law satisfactorily describes moisture diffusion in many situations, but that certain anomolies exist.[9,10] In studies of the effect of external stress, Gillat and Broutman found that external stress produces higher effective diffusivities.[11] This enhancement of the diffusion process can be attributed to the early formation of cracks through the thickness of 90° plys caused by moisture degradation of the strength of the transverse plys. Higher moisture contents decrease the after-drying and the wet short beam strength of the composite by enhancement of moisture introduction into the material. External stress accelerates the degradation of the interface- and matrix-dominated properties of the composite.

The dynamic response of cantilever beams of composite materials in a hygrothermal environment was discussed by Maymon, Briley, and Rehfield.[12] Dynamic characterization is very important because of vibration and flutter

TABLE A-2

LITERATURE SEARCH RESULTS

File	No. of Finds
NTIAC	54
NTIS	290
COMPENDEX	319
ISMEC	48
DDC	186
CHEMICAL ABSTRACTS	236
TOTAL	1133

requirements for many aerospace systems. Natural frequency and damping were determined for three different layups of graphite-epoxy materials at 93°C (200°F) with high relative humidity as well as dry at room temperature. The effective stiffness in the two layups having all or many 0°-fibers remained largely unaffected by environmental exposure because the behavior of the material was fiber dominated; however, for the ±45° specimens, the change in stiffness was appreciable but reversible. The effects of the hygrothermal environment on damping were appreciable for each layup, but damping increased for the all 0°-layup, yet decreased for the other two layups. The authors emphasized the possibility that hygrothermal environments could result in reduced stiffness and reduced damping occurring simultaneously, which could be deleterious from flutter or vibration considerations, or from both.

The effects of torsional and flexural fatigue on the long-time integrity of advanced graphite/epoxy structural composites subjected to environmental conditioning were investigated by Sumsion.[13] Results showed that in torsional cycling, both water environment and higher test temperature contributed to significant degradation of torsional stiffness. Flexural fatigue results on ±30° material showed a large fatigue effect with fatigue limits of 50% and 30% of the static failure strength. Compliance measurements indicated that the final failures were preceded by damage initiation and accumulation which began about 1% of the specimen life.

Investigations of the form of the moisture distribution on the fatigue behavior of graphite/epoxy composites have been reported by Ramani and Nelson.[14] For specimens having a large moisture gradient with an average moisture content of 1.4%, fatigue life was reduced by a factor of 8 at all stress levels investigated. Corresponding reduction in fatigue life for specimens having a flat moisture profile at the same average moisture content was comparatively smaller being about a factor 5 reduced from the value in dry specimens. X-ray radiographic analysis of damage accumulation in compression-compression fatigue revealed interlaminer cracking to be the dominant mode of failure responsible for the observed enhanced cyclic degradation of moisture condition specimens. This finding was corroborated by the observed systematic reduction in interlaminer shear strength as a function of moisture content which in turn increased the propensity for delamination under cyclic compressive loads. Residual strength measurements on cycled specimens indicated significant strength reductions had long lives, particularly in moisture condition specimens.

The majority of mechanical degradation studies reported in the literature are directed toward high performance graphite fiber reinforced composites or glass fiber reinforced composites where the environmental degradation has been shown to be primarily a matrix-dominated effect. In the case of a hygroscopic fiber such as Kevlar 49, the effects of moisture on mechanical properties may be expected to also be fiber-related. Studies of the absorption and transport of moisture in Kevlar 49 fiber have been reported

by Augl.[15] The purpose of the work was to measure the absorption equilibrium of Kevlar with moisture and its diffusion coefficient as a function of concentration. Kevlar 49 was found to have an unusually low diffusion coefficient compared to other polymers so that moisture transport in the composite is governed by the organic matrix or by other mechanisms such as microporosity. However, the equilibrium concentration as a function of relative humidity was found to be similar to most epoxy resins.

The effects of moisture and temperature on the flexural response of Kevlar reinforced epoxy laminates has been reported by Allred.[16] The author concludes that temperature or moisture alone did not severely affect the mechanical properties of Kevlar-epoxy, however, strength losses up to 60% and modulus losses of 45% were observed with saturation moisture contents (5 weight %) at 150°C. Results indicated that filament hygrothermal degradation was the dominant mechanism until moisture solvation depressed the matrix T_G below the test temperature. This is in direct contrast to the majority of high performance resin/matrix composites which exhibit little filament degradation.

NONDESTRUCTIVE DETECTION OF MOISTURE AND MECHANICAL DEGRADATION IN COMPOSITES

In the application of NDE technology to the measurement of moisture and determination of strength degradation in composite materials, several investigators have reported on the use of ultrasonics. In one study, Kaelble performed a study to determine whether sound velocity or attenuation measurements were directly sensitive to the state of the interfacial bond as evaluated by moisture absorption, interlaminer shear strength or fracture energy.[17] In experiments performed on graphite/epoxy specimens, Kaelble found that exposure to 95% relative humidity or water immersion at 100°C for times greater or less than 200 hours produced a 30 to 50% reduction in interlaminer shear strength, accompanied by a concurrent 2 to 5 fold increase in fracture toughness and acoustic absorption properties. These property changes were shown to be irreversible and directly related to cumulative moisture degradation of the fiber/matrix interfacial bond. The magnitude of the observed property changes were consistent with surface energy analysis and micromechanics predictions which show that fracture energy response optimizes at intermediate values of shear bond strength.

In experiments performed on a graphite/epoxy specimen subjected to length variable moisture exposure by partial submersion in a special container, Kaelble measured the longitudinal sound velocity C_1 and acoustic absorption coefficient α_1.[18] Results showed that the acoustic absorption coefficient α_1 remained relatively constant with position along the specimen, even though the moisture exposure history varied with position, except for cases where the specimen was exposed to thermal-shock cycles exceeding 177°C (350°F). The high attenuation was interpreted by the author in terms of internal cracking with visual evidence of internal delamination. Kaelble found that the ultrasonic velocity at 23°C and 2.25 MHz was very sensitive to moisture content.

A recent study of NDE of hygrothermal effects on fiber reinforced composites by ultrasonics has been reported by Bar-Cohen, et. al.[19] Carbon fiber reinforced laminates were immersed in room temperature water with some of the specimens being transferred to 90° water immersion for one day each week. At certain stages in the hygrothermal history, specimens were simultaneously tested destructively and ultrasonically. Because of the complexity of internal processes occurring simultaneously under hygrothermal conditions, it was found difficult to justify a direct correlation between strength data and transient nondestructive test measurements. However, results of the measurements showed that when the effects of variations in water absorption and desorption are deducted from the overall ultrasonic attenuation measurements, the net effect of material degradation may be correlated with variations in strength derived from destructive tests.

Applications of infrared thermography to study the effects of moisture in resin matrix composites have been reported by Singh, et al.[20] Several multiple graphite/polyimide composite specimens were examined by real/time infrared thermography to study the effect of moisture on their thermograms. Heat was injected from one side and the IR emission detected on the opposite side. No differences between the thermograms of dry and water containing specimens were detected for defect-free specimens. However, in defective specimens the presence of trapped moisture significantly modified the surface temperature distribution associated with regions of anomalous thermoconductance such as cracks, voids, and delaminations. Thus, a comparison between a thermogram of a defective test specimen as-received and its thermogram in a dry state can indicate if the test specimen has any moisture in it. The technique cannot, however, provide a quantitative measure of moisture content in the specimen.

The use of acoustic emission for studying environmental degradation of composites has been reported by Graham and Elsley.[21] Moisture degradation in graphite/epoxy composite specimens reduced the ultimate strength and resulted in a change in the acoustic emission amplitude distribution early in the loading history. Also, acoustic emission having distinct frequency spectral types tended to occur at or near singularities in the load curve. A computer pattern recognition technique was utilized to analyze the acoustic emission data in order to develop the statistical relationship between acoustic emission characteristics and mechanical strength parameters. The authors conclude that based upon the results obtained, acoustic emission provides a tool for methodically studying and identifying the specific failure modes which occur in composite materials under various environmental conditions.

Another approach which has been reported for monitoring strength degradation in graphite/epoxy composite material is moisture diffusion anlaysis.[22,23] In this approach, an electrolytic cell is utilized to record cumulative moisture evolution and moisture effusion rate. Variation of cell geometry accommodates either laboratory analysis of small composite

damaged control specimens or field inspection of limited surface areas of composite structure in a scanning mode. The moisture diffusion analysis approach provides information on current moisture content, directional diffusion coefficients and moisture concentration profile. The author indicates that studies which correlate moisture diffusion analysis with ultrasonic response and interlaminer shear strength of composite laminates subjected to localized moisture damage show that moisture diffusion analysis can be applied to map and locate low strength regions of a composite structure. Additional work is needed to quantify the method and generalize it to situations where anomalous diffusion occurs.

Radiofrequency spectroscopic approaches such as nuclear magnetic resonance (NMR) have also been applied for the detection of moisture and mechanical degradation in organic matrix composites. NMR is a method which provides a signal dependent on the number and characteristics of the hydrogen atoms in the material. The detected signals may be related not only to the amount of moisture present, but to the molecular structure involving hydrogen atoms. Investigators utilizing NMR to study the effects of water in epoxy resins and composite materials report the observation of at least two components to the NMR signal, one component associated with the hydrogen atoms in the rigid epoxy resin and the other component associated with the hydrogen atoms in the more mobile water.[24,25] Results reported thus far show that it is possible to independently measure the NMR characteristics associated with hydrogen in the epoxy and in the water.[25] This could prove useful in detecting and monitoring reversion because the relative number of mobile protons increases as reversion takes place. It would appear that NMR may also provide an approach for distinguishing moisture associated with the fiber/matrix interface from that diffusing through the resin matrix. Such results could prove useful in determining the relationship between moisture in various states of binding and mechanical property degradation.

REFERENCES

1. Broutman, L. J., Mazor, Abraham, and Eckstein, B. H., "Effect of Long Term Water Exposure on Properties of Carbon Fiber Reinforced Epoxies," Proceedings, National Technical Conference on High Performance Plastics, Society of Plastics Engineers, Oct. 1976.

2. Augl, J. M. and Trabocco, R., "Environmental Studies on Carbon Fiber Reinforced Epoxies," Proceedings, AFOSR Workshop on Durability Characteristics of Resin Matrix Composites, Battelle Memorial Institute, Columbus Laboratories, Columbus, Ohio, Oct. 1975.

3. McKague, E. L., Reynolds, J. D., and Halkias, J. E., "Life Assurance of Composite Structures, Volume 1, Moisture Effects," Technical Report AFML-TR-75-51. Air Force Materials Laboratory, Dayton, Ohio, 1975.

4. Browning, C. E., Husman, G. E., and Whitney, J. M. in Composite Materials: Testing and Design (Fourth Conference), ASTM STP 617, American Society for Testing and Materials, pp. 481-496, 1977.

5. Shen, Chi-Hung and Springer, G. S., Journal of Composite Materials, Vol. II, pp. 2-16, 1977.

6. DeIasi, R. and Whiteside, J. B., "Effect of Moisture on Epoxy Resins and Composites," Advanced Composite Materials-Environmental Effects, ASTM STP 658, J. R. Vinson, Ed., American Society for Testing and Materials, pp. 2-20, 1978.

7. Shen, Chi-Hung and Springer, G. S., Journal of Composite Materials, Vol. 10, pp. 2-20, Jan. 1976.

8. Advanced Composite Materials - Environmental Effects, ASTM STP 658, American Society for Testing and Materials, Philadelphia, 1978.

9. Shirrell, C. D., "Diffusion of Water Vapor in Graphite/Epoxy Composites," Advanced Composite Materials-Environmental Effects, ASTM STP 658, J. R. Vinson, Ed., American Society for Testing and Materials, pp. 21-42, 1978.

10. Whitney, J. M. and Browning, C. E., "Some Anomalies Associated with Moisture Diffusion in Epoxy Matrix Composite Materials," Advanced Composite Materials-Environmental Effects. ASTM STP 658, J. R. Vinson, Ed., American Society for Testing and Materials, pp. 43-60, 1978.

11. Gillat, O. and Broutman, L. J., Effect of an External Stress on Moisture Diffusion and Degradation in a Graphite-Reinforced Epoxy Laminate," Advanced Composite Materials-Environmental Effects, ASTM STP 658, J. R. Vinson, Ed., American Society for Testing and Materials, pp. 61-83, 1978.

12. Maymon, G., Briley, R. P., and Rehfield, L. W., "Influence of Moisture Absorption and Elevated Temperature on the Dynamic Behavior of Resin Matrix Composites: Preliminary Results," Advanced Composite Materials-Environmental Effects. ASTM STP 658, J. R. Vinson, Ed., American Society for Testing and Materials, pp. 221-233, 1978.

13. Sumsion, H. T., "Environmental Effects on Graphite-Epoxy Fatigue Properties," J. Spacecraft and Rockets, 13, No. 3, pp. 150-155, 1976.

14. Ramani, S. V. and Nelson, H. G., "Effect of Moisture on the Fatigue Behavior of Graphite/Epoxy Composite Laminates," Report No. NASA-TM-78548, Amer. Research Center, CA, pp. 1-49, 1979.

15. Augl, J. M., "Moisture Sorption and Diffusion in Kevlar 49 Aramid Fiber," Report No. NSWC/TR-79-51, Naval Surface Weapons Center, White Oak Lab, Silver Spring, MD, pp. 1-48, 1979.

16. Allred, R. E., "Hygrothermal Effects on the Mechanical Response of Kevlar 49/Epoxy Laminates," Report No. CONF-781067-4, Sandia Laboratories, Albuquerque, NM, pp. 1-16, 1978.

17. Kaelble, D. H., "Detection of Hydrothermal Aging in Composite Materials," Proc. of ARPA/AFML Rev. of Quant. NDE, AFML-TR-75-212, pp. 549-564, 1976.

18. Kaelble, D. H. and Dynes, P. J., "Methods for Detecting Moisture Degradation in Graphite-Epoxy Composites," Materials Evaluation, 35, No. 4, pp. 103-108, 1977.

19. Bar-Cohen, Y., Meron, M., and Ishai, O., "Nondestructive Evaluation of Hygrothermal Effects on Fiber-Reinforced Plastic Laminates," J. Testing and Eval., 7, No. 5, pp. 291-296, 1979.

20. Singh, J. J., Kantsios, A. G., Moerlean, E. A., Babcock, R. A., and Buckingham, R. D., "Effects of Moisture in Infrared Thermography of Resin Matrix Composites," Report No. NASA-TM-78649, Langley Research Center, Langley Station, VA, pp. 1-18, 1978.

21. Graham, L. J. and Elsley, R. K., "Characteristics of Acoustic Emission Signals from Composites," Proc. of ARPA/AFML Rev. of Prog. in Quant. NDE, AFML-TR-78-55, pp. 219-225, 1978.

22. Kaelble, D. H., "Moisture Diffusion Analysis of Composite Strength Degradation," Proc. of ARPA/AFML Rev. of Prog. in Quant. NDE, AFML-TR-78-55, pp. 226-232, 1978.

23. Kaelble, D. H., "Wetometer for Measurement of Moisture in Composites," Proc. of ARPA/AFML Rev. of Prog. in Quant. NDE, AFML-TR-78-55, pp. 371-376, 1978.

24. King, J. D., Rollwitz, W. L. and Matzkanin, G. A., "Magnetic Resonance Methods for NDE," Proc. 12th Symposium on NDE, pp. 138-149, 1979.

25. Brown, I. M., and Lind, A.C., "Study of the Glass Transition in Crosslinked Polymers Using Nuclear and Electron Magnetic Resonance", Final Report, Contract No. 00019-76-C-0565, Naval Air Systems Command, 1977.

APPENDIX B

NMR MOISTURE MEASUREMENT

Basic Principles of NMR

Nuclear magnetic resonance is a branch of spectroscopy based on the interaction between nuclear magnetic dipole moments and a magnetic field. The term resonance is used because a natural frequency of the magnetic system, namely, the frequency of gyroscopic precession of the magnetic moments in an applied static magnetic field, is detected. In the following paragraphs, the basic principles of NMR are briefly described; the fundamentals of NMR are explained in great detail in several texts.[1-3]

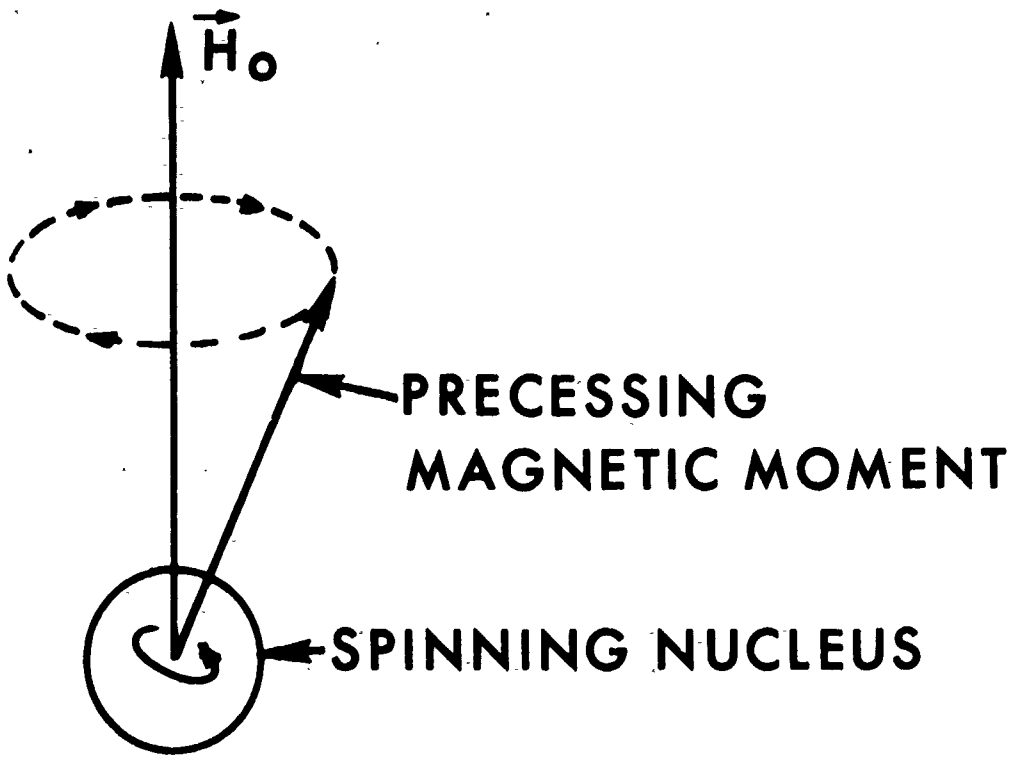
Many atomic nuclei possess a non-zero spin angular momentum and an associated magnetic dipole moment. Analogous to the precession of a spinning top in a gravitational field, the spinning nuclear magnet precesses about the direction of an applied steady magnetic field, $\vec{H}_o$, as depicted in Figure B1. The angular precession frequency is directly proportional to the strength of the magnetic field with the proportionality constant (called the gyromagnetic ratio) having a value characteristic of the specific nuclear species. For the hydrogen nucleus, the gyromagnetic ratio has the value 2.67×10^4 $(G\text{-}sec)^{-1}$. A "resonance" condition can be achieved by application of a second magnetic field alternating at a frequency corresponding to the precessional frequency of the nuclei of interest, and in a direction orthogonal to that of the steady magnetic field. For appropriate values of steady and alternating magnetic fields, the spin axis of the nucleus can be reoriented, and this is the phenomenon of nuclear magnetic resonance.

Several methods of generating and detecting NMR signals are available. The method preferred for most practical applications is the transient or pulsed method because measurements can be made rapidly and the data obtained provide much information about the investigated nuclear species. In this approach, the RF field is applied in short bursts or pulses. The magnitude and duration of the RF pulse determines through what angle with respect to the static magnetic field the nuclear magnetization vector will be reoriented during the pulse. In general, the angle through which the magnetization, $\vec{M}_o$, is rotated is given by

$$\theta = \gamma H_1 \tau_w$$

where H_1 is the amplitude of the rotating RF magnetic field and τ_w is the duration of the pulse. The particular pulses most commonly utilized in transient NMR experiments are those for which $\theta = 90°$ (90° pulse) or $\theta = 180°$ (180° pulse). These are illustrated schematically in Figure B2.

The pulsed NMR approach affords a direct means for measuring the nuclear magnetization, $\vec{M}_o$. For example, at the termination of a 90° pulse, the magnetization lies in the plane perpendicular to $\vec{H}_o$, and its precession about $\vec{H}_o$ in that plane is capable of inducing a measurable voltage in the RF coil which then serves as a sensor. The magnitude of this voltage is proportional to the number of nuclei within the effective volume of the coil. Of basic interest is the lifetime of the signal induced by the nuclear precession. Two effects must be taken into consideration: (1) interactions between the nuclei in the specimen tend to diminish the amplitude of the precessing transverse magnetization, and (2) inhomogeneities



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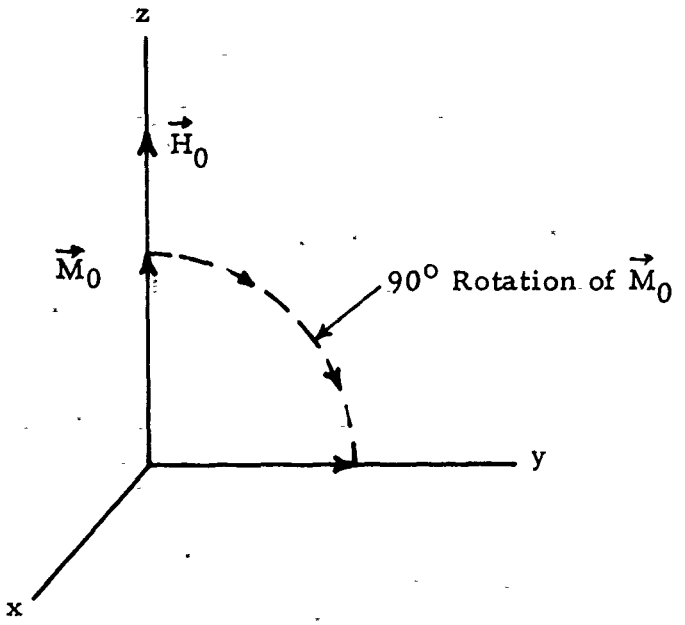
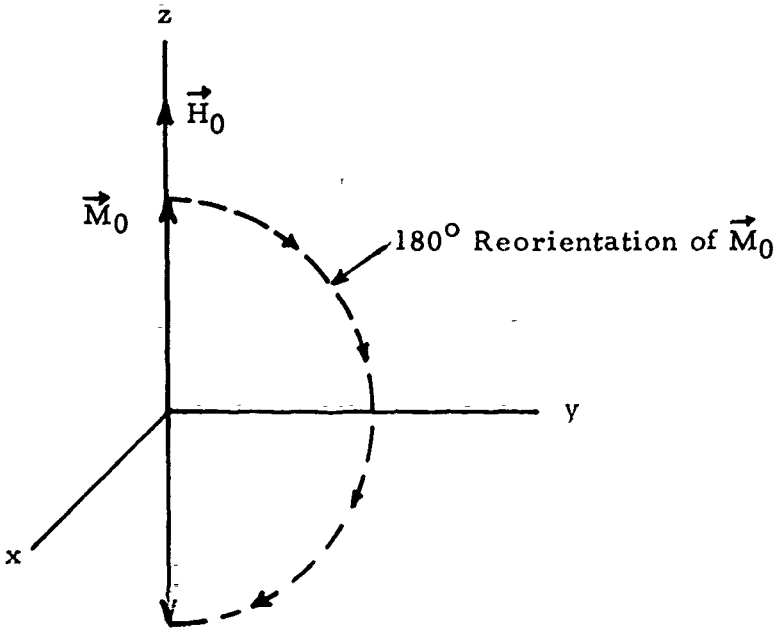
PRECESSION FREQUENCY $\propto$ MAGNETIC FIELD

FIGURE B1. PRECESSION OF A NUCLEAR MAGNETIC MOMENT IN A STATIC MAGNETIC FIELD



a. Reorientation of Nuclear Magnetization by a 90° Pulse

3041



b. Reorientation of Nuclear Magnetization by a 180° Pulse

FIGURE B2. SCHEMATIC REPRESENTATION OF NUCLEAR MAGNETIZATION REORIENTATIONS BY PULSED NMR

in the applied static field gradually destroy the coherence among the individual nuclear spins composing the net magnetization causing a "fanning out" of the precessing nuclear magnetic moments. These effects are illustrated schematically in Figure B3. As a consequence of these two effects, the voltage signal induced by the precessing magnetization gradually decays to zero; this is called "free induction decay" and is illustrated schematically in Figure B4. If now a second RF pulse is applied to the specimen at a suitable time following the first pulse, namely, after the initial voltage has decayed because of the "fanning out" of the nuclear magnetic moments, but before the amplitude of the transverse magnetization has itself diminished to zero due to internal interactions, then the nuclear magnetic moments can be refocussed into a "spin echo", which manifests itself as an induced voltage following the second applied pulse. This phenomenon is depicted schematically in Figure B5 and examples of hydrogen spin echo signals obtained from mixtures of bentonite and water are shown in Figure B6.

The amplitude and shape characteristics of the free induction decay and spin echo signals provide a variety of information about the specimen including (1) the presence of atoms in different physical states (e.g. liquid and solid); (2) the type and strength of interactions between nuclei of both the same species and different species; and (3) changes in molecular structure and intermolecular binding.

In real macroscopic specimens, the nuclear magnetic moments are not free but interact with each other and with their surroundings. As a consequence, following application of a 90° pulse the nuclear magnetization does not return to its equilibrium value along the magnetic field instantaneously but rather does so over a period of time. Since the interactions responsible for alignment of the nuclear magnetization along the magnetic field are associated with energy exchange between the nuclear spins and the lattice, the recovery time is called the spin-lattice relaxation time, T_1. This relaxation time may be measured by application of a sequence of two 90° pulses. If the amplitude of the free induction decay following the second pulse is measured as a function of the spacing between the pulses, the recovery time of the spin system may be determined.

In addition to the interactions between the nuclei and the lattice which are responsible for spin-lattice relaxation, transverse components of the nuclear magnetization (generated, for example, by a 90° pulse) represent a non-equilibrium situation and decay due to spin-spin interactions. These interactions are responsible for the decay of the transverse magnetization depicted earlier in Figure B3. In solids, the spin-spin relaxation time, T_2 is generally quite short due to strong spin-spin interactions. However, for more loosely bound nuclei, such as in liquids, spin-spin interactions are reduced by molecular motion resulting in a lengthened relaxation time, T_2. If the spin-spin relaxation time of the nuclear spin system (i.e., the natural decay time of the transverse magnetization) is shorter than the spin de-phasing caused by an inhomogeneous magnetic field (see Figure B3), then T_2 may be determined directly from the free induction decay. For longer T_2's, the spin-spin relaxation times may be determined by application of a 90^o-180^o pulse sequence and measuring the amplitude of the spin echo following the 180^o pulse. Because of the shorter relaxation time for solids compared with liquids, the spin echo

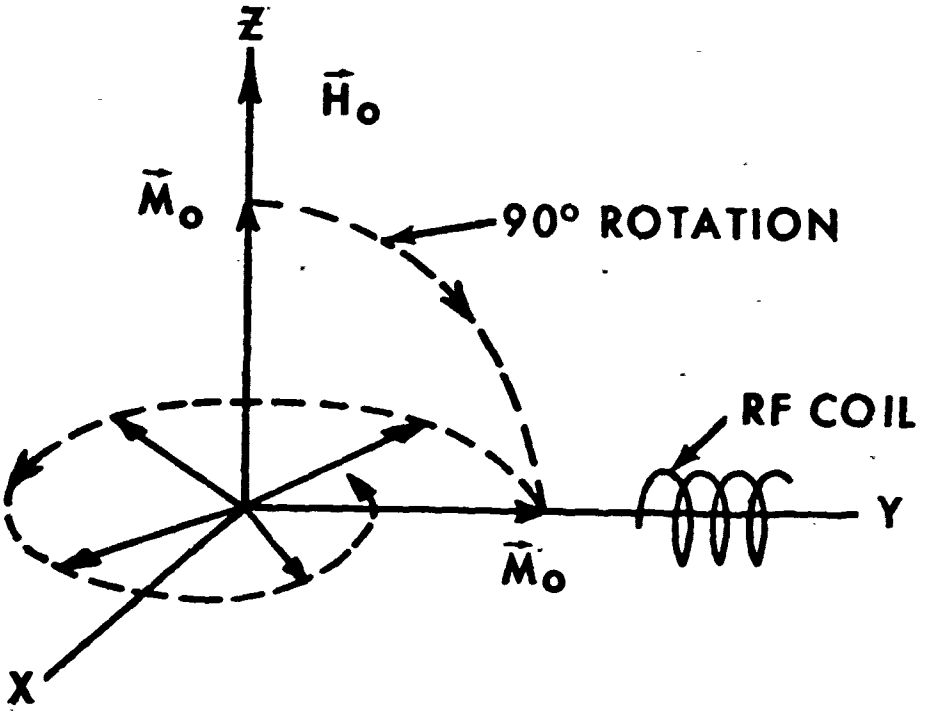


FIGURE B3. 90° ROTATION OF THE NUCLEAR MAGNETIZATION BY AN RF PULSE, FOLLOWED BY FREE INDUCTION DECAY

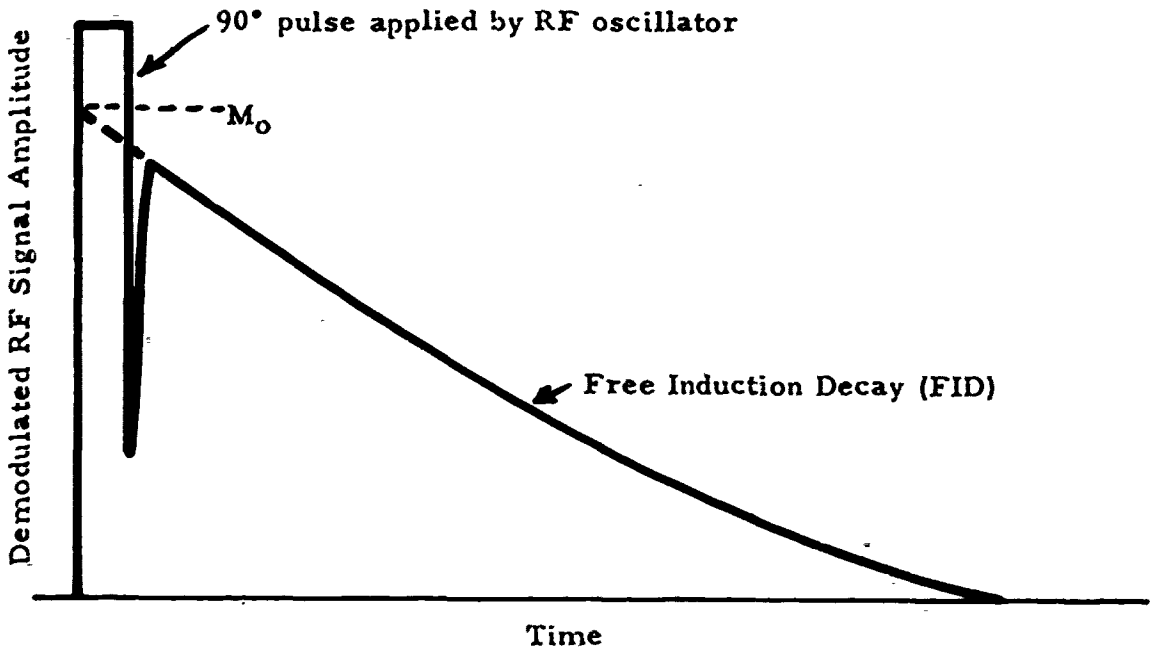


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FIGURE B4. SCHEMATIC REPRESENTATION OF THE FREE INDUCTION DECAY SIGNAL FOLLOWING A 90° PULSE

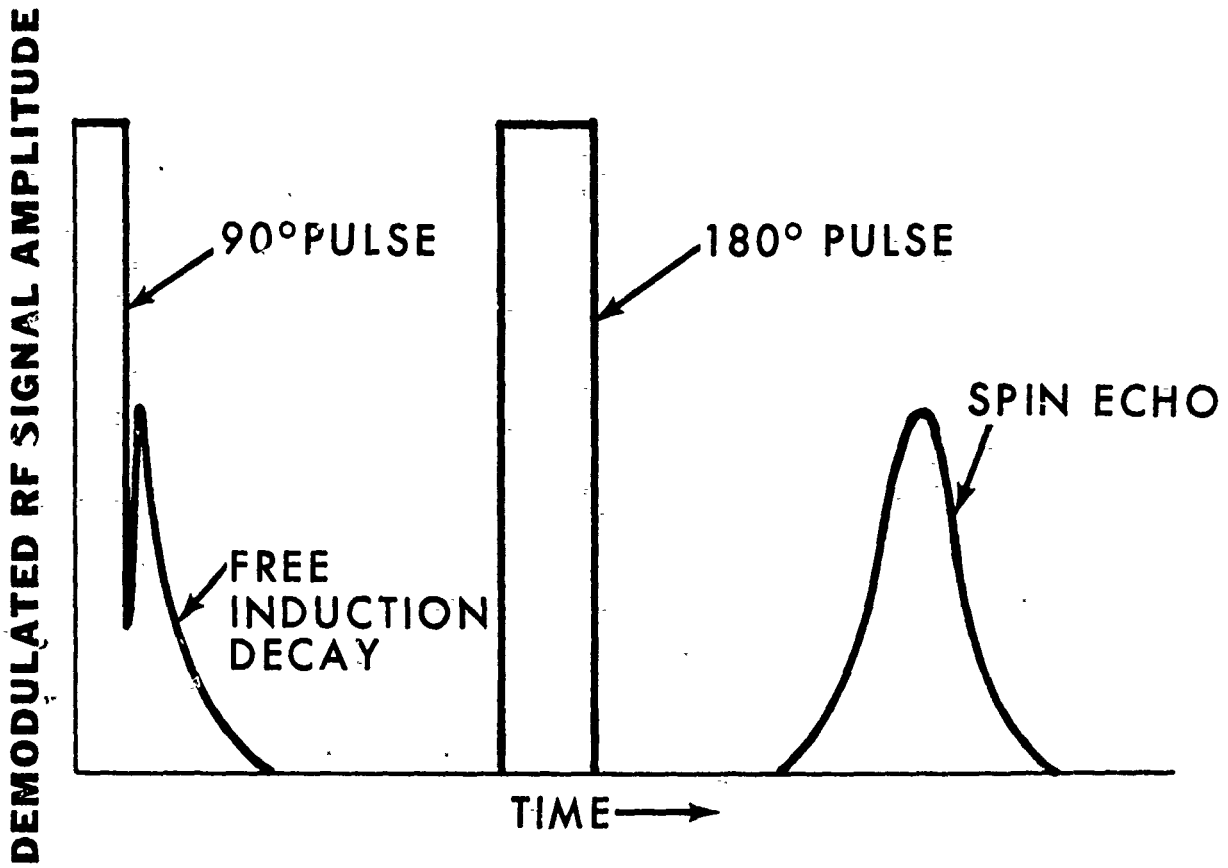
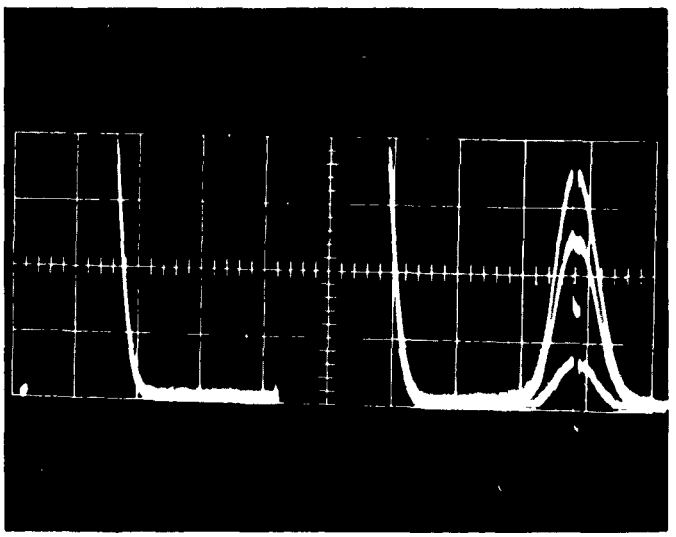


FIGURE B5. SCHEMATIC DIAGRAM OF A FREE INDUCTION DECAY AND SPIN ECHO ASSOCIATED WITH A TWO-PULSE SEQUENCE

3021



Vertical: .5 V/cm
Horizontal: 50 μs/cm
Upper Echo: 100% H_2O
Middle Echo: 50% H_2O
Lower Echo: 20% H_2O

FIGURE B6. OSCILLOSCOPE PHOTOGRAPH OF TYPICAL PULSED NMR SPIN ECHOES FROM MOISTURE IN BENTONITE

technique provides a means for separately determining signal contributions from liquid components. Thus, if the time interval between the successive 90° and 180° pulses is adjusted to be greater than the T_2 associated with the nuclei in the solid state, but less than the T_2 associated with the nuclei in the liquid constituent, then the transverse magnetization associated with the solid will have decayed away before the 180° pulse is applied, while that portion of the transverse magnetization resulting from nuclei in the liquid constituent though de-phased, will not have appreciably diminished in magnitude, and will be refocussed into a spin echo signal by the 180° pulse. Thus, the spin echo approach provides a method for detecting only the NMR signal due to the liquid component when both liquid and solid constituents possessing a common nucleus are present in a specimen.

The essential elements of the apparatus used to implement the pulsed NMR method are shown schematically in Figure B7. The specimen (1) is encircled by a radiofrequency induction coil (2). A static magnetic field of strength H_o is applied by a magnet (3). A radiofrequency oscillator (4) supplies pulses of RF energy to the coil of the proper duration and separation to produce free induction decay or spin echo signals. The signal voltage induced in the sensor coil is amplified by the RF amplifier (5), demodulated by the detector (6), and displayed as a function of time on a cathode ray oscilloscope (7). A digital readout of the amplitude of the signal is implemented by a sampling gate (8) and a digital voltmeter (9).

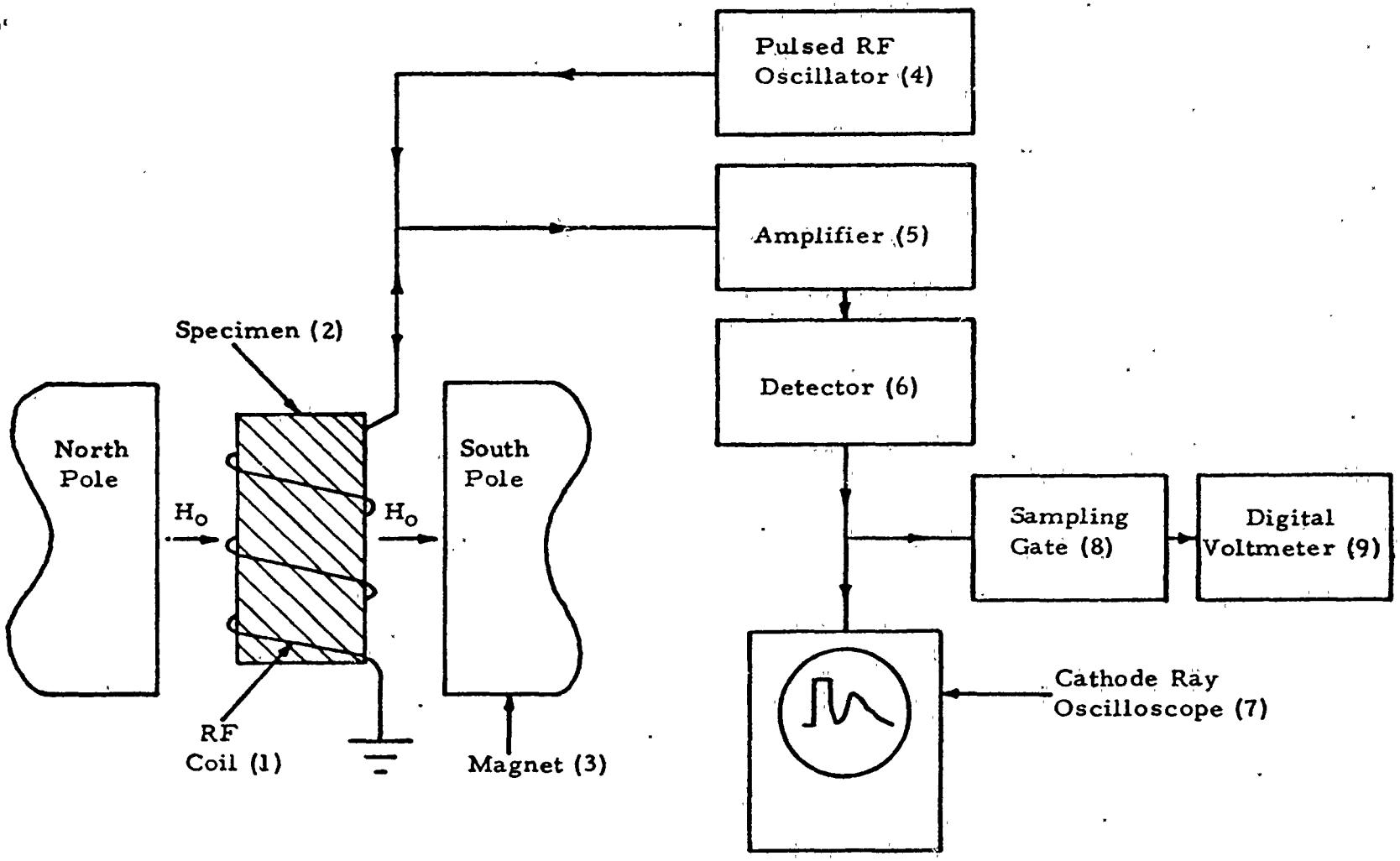


FIGURE B7. ESSENTIAL ELEMENTS OF LABORATORY ARRANGEMENT FOR PULSED NMR EXPERIMENTS (Schematic)

LIST OF REFERENCES

1. Abragam, A., The Principles of Nuclear Magnetism, Clarendon Press, Oxford, 1961.

2. Slichter, C. P., Principles of Magnetic Resonance, Harper Row, New York, 1963.

3. Andrew, E. R., Nuclear Magnetic Resonance, Cambridge University Press, Cambridge, England, 1955.

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INVESTIGATION OF THE EFFECT OF MOISTURE ON THE MECHANICAL PROPERTIES OF ORGANIC MATRIX COMPOSITE MATERIALS USING NUCLEAR MAGNETIC RESONANCE -
G. A. Matzkanin, Southwest Research Institute,
6220 Culebra Road, P.O. Drawer 28510,
San Antonio, Texas 78284

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